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Volume 29 MARCH, 1959 Number 1

Journal of Sedimentary Petrology

A Publication of the Society of Economic Paleontologists and Mineralogists
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The American Association of Petroleum Geologists

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Journal
of
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VOLUME 29
MARCH, 1959-DECEMBER, 1959
NOS. 1-4

J. L. HOUGH, EDITOR

PUBLISHED BY THE
SOCIETY OF ECONOMIC PALEONTOLOGISTS AND
MINERALOGISTS
A DIVISION OF
THE AMERICAN ASSOCIATION OF PETROLEUM GEOLOGISTS
Box 979, Tulsa, Oklahoma

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The *Journal of Sedimentary Petrology* is published in March, June, September, and December of each year.

Annual membership dues of the Society of Economic Paleontologists and Mineralogists are as follows:

including the Journal of Sedimentary Petrology	$ 7.00
including the Journal of Paleontology	10.00
including both journals	17.00

The subscription prices of the journals, to non-members of the Society, are as follows:

Journal of Sedimentary Petrology	$ 9.00
Journal of Paleontology	15.00

Postage is included in these prices, for all addresses in the United States. An additional charge of 40 cents on annual subscriptions is made for addresses in all other countries.

The price of single numbers of the *Journal of Sedimentary Petrology* is $1.50 to members and $3.00 to non-members of the Society.

Communications about *Journal* subscriptions, rates, memberships, change of address, and non-receipt of preceding number should be addressed to Society of Economic Paleontologists, and Mineralogists, P.O. Box 979, Tulsa, Oklahoma. Claims for non-receipt of preceding numbers must be sent in within three months of the date of publication in order to be filled gratis.

Communications regarding manuscripts and purely editorial matters should be addressed to The Editor, *Journal of Sedimentary Petrology*, Department of Geology, University of Illinois, Urbana, Illinois.

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Articles submitted for publication in the *Journal of Sedimentary Petrology* should be original pages, typewritten double-spaced on 8½ by 11-inch white paper. A prefatory abstract is required for all papers, exclusive of short notes and discussions. The mechanical details of the manuscript should conform with usage in recent issues of the *Journal*, particularly in regard to capitalization and citation of references. Tables and illustrations should be separate from the text, and originals of illustrations are preferred. Detailed instructions and suggestions are given in the March, 1958 issue of the *Journal* (v. 28, p. 102–107).

Second-class postage paid at the Post Office at Menasha, Wisconsin.

GEORGE BANTA COMPANY, INC., MENASHA, WISCONSIN,
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EVIDENCE OF TRANSGRESSIVE-REGRESSIVE CAMBRIAN SANDSTONES BORDERING LAKE SUPERIOR[1]

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ABSTRACT

The Upper Cambrian Munising Sandstone bordering the southern shore of Lake Superior is divided into 3 members; the basal conglomerate, Chapel Rock Member, and Miner's Castle Member. Heavy mineral similarities indicate that the Chapel Rock Member may be correlated with the Dresbach of Wisconsin. The Miner's Castle Member disconformably overlies the Chapel Rock Member. Variations in sorting and mean grain diameter through the Miner's Castle Member indicate transgressive and regressive depositional environments. Variations in the heavy mineral suite, particularly in garnet content, indicate that the Miner's Castle Member is transitional between the transgressive Franconia Sandstone and the regressive Jordan Sandstone.

INTRODUCTION

Correlation and dating of the sandstones bordering the southern shore of Lake Superior are difficult due to the unfossiliferous nature of the beds. The Munising Sandstone overlies the Jacobsville Sandstone in the cliffs bordering the lake and is, in turn, overlain by the Au Train Formation. In 1951 Oetking concluded primarily on the basis of heavy mineral studies that both the Dresbach and the Franconia of the Wisconsin section are present in the Munising Sandstone. By using paleontologic evidence he determined the age of the overlying Au Train Formation to be lower Middle Ordovician and correlated it with the Plattville Formation of Wisconsin. Previously the Au Train had been dated as Upper Cambrian or Lower Ordovician. Hamblin (1958) also correlated the Munising Sandstone with the Dresbach and Franconia of the Wisconsin section.

The present study was made in part to examine Oetking's proposal (1951, p. 33) that "the Munising Sandstone group could be subdivided into the Dresbach and Franconia formations of Croixan age on the basis of lithology, heavy mineral suites, and the stratigraphic position in the geologic column, . . . " and in part to better define the relationship of the Munising beds to the Croixan section in southern Wisconsin. Detailed heavy mineral and size analyses of the Munising Sandstone have provided evidence requiring an interpretation somewhat at variance with that of previous workers. Field studies were made during the summer of 1953 and part of the summer of 1955 between Marquette and Grand Marais, Michigan.

LABORATORY METHODS

Mechanical analysis of size grades coarser than $\frac{1}{16}$ mm was made with a standard set of Tyler sieves which were shaken in a Rotap shaker for a period of 10 minutes. From the cumulative grain size distribution curves, phi mean diameter and phi deviation were estimated. Estimates were made as suggested by Inman (1951).

Tetrabromethane was used in all heavy mineral separations. The $\frac{1}{8}$ mm to $\frac{1}{16}$ mm grade, or a representative fraction thereof, was selected for analysis. The heavy fraction of this grade was mounted in permanent mounts and 200 grains were counted on each slide.

Authigenic pyrite occasionally constitutes a large part of the heavy fraction. It was excluded from the calculations employed in the construction of figures 3 and 4 but tables 1 through 5 show percent by number of heavy minerals including authigenic pyrite.

STRATIGRAPHY

Following the suggestion of Hamblin (1958, p. 71) the Munising Sandstone is here divided into three members; the basal conglomerate, the Chapel Rock Member, and the Miner's Castle Member.

Numerous exposures of the Munising Sandstone are found between Marquette

[1] Manuscript received June 17, 1958.

TABLE 1.—*Lithology, heavy mineral frequency, and grain size parameters through Mosquito Harbor section*

Lithology	Thickness in Feet	Percent Frequency by Number							Grain Size Parameters	
		Garnet	Zircon	Leucoxene	Ilmenite	Pyrite	Magnetite	Tourmaline	$M\phi$	$\sigma\phi$
Munising Sandstone										
Miner's Castle Member										
6. Sandstone with occasional shale lenses, white or yellow, heavily iron stained, bedding average 6 inches.	4.0	9	7	11	6	30	2	6	1.82	0.36
5. Sandstone, white, weathers yellow or orange, poorly cemented, bedding averages 5 feet. Inaccessible. Chapel Rock-Miner's Castle contact is within this unit.	25±									
Chapel Rock Member										
4. Sandstone, white, iron stained in upper part, bedding averages 3 feet.	7.5	—	9	21	5	7	7	6	1.85	0.73
3. Sandstone, white, weathers yellowish brown, bedding $\frac{1}{4}$ inch to 2 inches. Small black granules scattered throughout. Concretions of sandstone strongly cemented with iron oxide scattered throughout. These "rock roses" average 1.5 inches in diameter.	5.5	—	3	26	3	15	1	<1	0.88	1.14
		—	9	33	10	18	9	6	1.87	0.78
2. Shale, small lenses of sand, black, hard, laminations average $\frac{1}{4}$ inch thick, current ripple marks well developed.	0.2									
1. Conglomerate, matrix white, weathers yellow, red or brown, cobbles up to 8 inches in diameter, average 2 inches. Upper part has no cobbles but shows current ripple marks and mud cracks.	2.3	1	8	18	20	3	7	5		

and Grand Marais, Michigan. Those at Miner's Castle, Laughing Whitefish Falls, Munising Falls, and Chapel Falls are of particular interest since all show nearly the entire thickness of the Miner's Castle Member, and the section at Miner's Castle contains nearly the entire thickness of the Munising Sandstone.

The unweathered Munising Sandstone is white, light gray, or light brown. Cliff exposures like that of the Pictured Rocks are varigated and vertically striped with brilliant yellows and reds owing to the deposition of iron oxides from water seeping from the cliffs.

The characteristics of the Munsing Sandstone are much more uniform throughout the area than those of the underlying Jacobsville Sandstone which is extremely variable in color, grain size, sorting and bedding. Bedding in the Munising ranges from a few inches to several feet. Crossbedding is particularly common in the Chapel Rock Member.

BASAL CONGLOMERATE

The basal conglomerate of the Munising Sandstone is best exposed in the shore cliffs of Grand Island but is also found at Mosquito Harbor, $SE\frac{1}{4}$ $SE\frac{1}{4}$ sec. 35, T. 48 N.,

TABLE 2.—*Lithology, heavy mineral frequency, and grain size parameters through Miner's Castle section*

Lithology	Thickness in Feet	Percent Frequency by Number						Grain Size Parameters	
		Garnet	Zircon	Leucoxene	Ilmenite	Pyrite	Tourmaline	Mϕ	σϕ
Munising Sandstone									
Miner's Castle Member									
11. Sandstone, white, some layers heavily iron stained, bedding 1 foot to 4 feet, averages 3 feet.	13	93	—	3	1	—	1	1.75	1.03
10. Sandstone, some shale in upper part, white, varigated weathering, poorly cemented, bedding up to 7 feet, averages 1 foot, occasional cross-bedding.	23	93	2	5	1	—	—	1.60	0.87
		88	—	6	<1	—	2	1.60	0.99
		61	2	20	6	<1	4	0.47	0.77
9. Shale, arenaceous, gray.	0.2	6	—	12	<1	80	—	2.65	1.51
8. Sandstone, thin shale layers, white, weathers yellow and gray, occasional cross-bedding.	4.2	79	2	8	4	1	6	1.75	0.89
7. Shale, arenaceous, gray, strongly cemented.	0.7	13	3	23	—	57	—	—	—
6. Sandstone with minor shale, white or gray, weathers yellow to rust color, poorly cemented, bedding 0.5 to 8 inches, occasional cross-bedding.	8.5	16	1	21	—	58	2	1.82	1.07
5. Sandstone with minor shale, weathers light to bright yellow, occasional large pebbles, iron stained beds in lower part, bedding 1 inch to 5 feet, averages 1.5 feet.	34.1	31	4	40	2	3	10	1.48	1.10
		75	2	3	2	<1	10	1.81	1.33
		76	1	8	3	<1	4	2.11	1.07
4. Sandstone with minor shale, pure white, poorly cemented, thin bedded, cross-bedding prominent.	8.3	67	3	4	2	12	8	2.27	1.09
3. Sandstone, red brown to black, speckled with orange, lighter on weathered surfaces, occasional cross-bedding.	3.1	11	18	27	4	24	6	2.07	1.03
Chapel Rock Member									
2. Shale, dark gray to black, mudcracks present on west side of Miner's Castle turrets.	0.1								
1. Sandstone, pink, light orange or white, darker toward top, bedding averages 2 inches near base, 10 inches near top, cross-bedding prominent.	16.9	6	22	36	11	4	4	1.84	0.67
		6	33	28	12	2	12	1.74	0.97

R. 18 W. and Au Sable Falls, NW¼ NW¼ sec. 2, T. 49 N., R. 14 W. The conglomerate is 1 to 12 feet thick and consists of cobbles up to 8 inches in diameter of quartz, quartzite, sandstone, clay, and acid and basic igneous rocks set in a sand matrix. At Au Sable Falls occasional cobbles are found well up in the Chapel Rock Member, and are

TABLE 3.—*Lithology, heavy mineral frequency, and grain size parameters through Laughing Whitefish Falls section*

Lithology	Thickness in Feet	Percent Frequency by Number							Grain Size Parameters	
		Garnet	Zircon	Leucoxene	Ilmenite	Pyrite	Tourmaline	Hematite	Mϕ	σϕ
Munising Sandstone										
Miner's Castle Member										
3. Sandstone, white, weathers red, occasional thin iron stained layers, bedding 1 foot to 4 feet, averages 1.5 feet, cross-bedding common in some beds, glauconite present in thin bed 2.5 feet above base of unit.	22.0	86	—	2	3	5	<1	—	1.39	0.87
		98	<1	1	<1	—	—	—	1.49	0.73
		86	1	3	<1	9	—	1	1.49	0.75
2. Sandstone, red to pink, darker on weathered surfaces, poorly cemented with iron oxide.	1.2	83	—	17	—	—	—	—	1.40	0.70
1. Sandstone and shale, shale 30% at base, gradually disappearing toward top. Sandstone is white to gray showing glauconite and iron staining. Shale is gray with occasional glauconite staining. Bedding 0.5 inches to 8 inches.	71.0	79	—	4	—	16	<1	—	1.67	0.74
		15	<1	<1	<1	83	—	—	1.75	1.11
		47	—	<1	1	50	1	—	2.19	1.13
		33	<1	1	<1	64	—	—	1.97	1.25
		23	<1	4	1	69	<1	—	1.85	1.24
		22	—	10	<1	66	<1	—	2.71	1.39
		42	2	25	4	26	<1	—	2.71	1.44
		39	1	38	6	10	3	1	1.98	1.18
		18	3	48	7	15	6	—	2.25	1.38
		8	4	61	12	5	2	3	2.01	1.17
		11	1	62	11	4	1	3	2.17	1.37
		14	1	59	12	1	3	4	2.03	1.11
		18	4	35	12	3	7	9	2.53	1.41

not strictly limited to the basal conglomerate as in other outcrops.

A pure white, well sorted, and poorly consolidated sandstone sporadically occurs below the conglomerate and above the Jacobsville sandstone. The sandstone has a thickness of 4 feet in the cliffs of Grand Island where it shows prominent cross-bedding. At Mosquito Harbor current ripple marks and mud cracks occur directly above the conglomerate.

Chapel Rock Member

At Mosquito Harbor a nearly complete section of the Chapel Rock Member is exposed. It is overlain by the Miner's Castle but this member is largely inaccessible for sampling at this locality due to the steepness of the cliff face on which it is exposed. Table 1 shows the lithology, the heavy mineral analyses, and size analyses of this section. At Miner's Castle, SW¼ SW¼ sec. 3, T. 47 N., R. 18 W. a section of the major part of the Chapel Rock Member is exposed beneath the Miner's Castle Member. The basal conglomerate is beneath lake level at this locality. The Au Train Formation is exposed above the Miner's Castle Member in the turrets of Miner's Castle. The large scale cross-bedding commonly found in the Chapel Rock Member is well exposed in this outcrop. Table 2 shows the lithology, and the heavy mineral and size analyses of the Munising at this locality.

Miner's Castle Member

At Miner's Castle mudcracks at the top of the Chapel Rock Member indicate sub-

TABLE 4.—*Lithology, heavy mineral frequency, and grain size parameters through Munising Falls section*

Lithology	Thickness in Feet	Percent Frequency by Number						Grain Size Parameters	
		Garnet	Zircon	Leucoxene	Ilmenite	Pyrite	Tourmaline	Mϕ	σϕ
Au Train Formation									
6. Sandy dolomite, yellow to light gray.	1.5								
Covered interval	4.0								
Munising Sandstone									
Miner's Castle Member									
5. Sandstone, white with occasional iron stained layers, weathers white or gray, streaked with orange, poorly cemented, bedding 1 foot to 6 feet, averages 2 feet, cross-bedding prominent.	35.5	98	—	2	—	—	1	1.20	0.73
		93	—	3	<1	—	<1	1.37	0.77
		91	—	3	<1	1	—	1.24	0.73
		96	—	3	<1	—	—	1.28	0.73
		87	<1	8	1	<1	1	1.50	0.87
		90	2	4	—	1	2	1.77	0.94
Covered interval	6.5								
4. Sandstone, white, weathers gray brown, carbonate cement abundant, bedding 2 inches to 4 feet, averages 1 foot.	7.3	32	—	4	—	57	1	1.87	1.19
3. Sandstone, white, occasionally iron stained, weathers deep brown, poorly cemented with carbonate, bedding 1 foot to 5 feet, averages 4 feet.	12.5	65	1	7	1	21	—	2.19	1.45
		57	—	4	1	35	<1	1.69	1.31
2. Sandstone, white, occasionally iron stained, weathers yellow brown, carbonate cement abundant, bedding 0.5 inches to 5 inches, averages 2 inches, very thin occasional shale laminae.	12.5	72	1	10	2	9	<1	1.84	1.30
		31	1	22	1	36	3	2.27	1.13
Covered interval	12.0								
1. Sandstone, white, weathers light yellow, massive bedding, cross-bedding prominent, occasional thin laminae of gray shale.	7.5	44	4	4	3	32	8	1.57	1.09

aerial exposure prior to the deposition of the Miner's Castle Member. Excellent exposures of the Miner's Castle also were studied at Laughing Whitefish Falls, C. N½ SW¼ sec. 16, T. 46 N., R. 22 W., Munising Falls, SW¼ NE¼ sec. 31, T. 47 N., R. 18 W., and Chapel Falls, NE¼ SW¼ sec. 28, T. 48 N., R. 17 W. The lithology, and heavy mineral and size analyses of these sections are given in tables 3, 4, and 5 respectively.

Only in the Munising Falls and Miner's Castle sections was the Au Train Formation exposed above the Munising Sandstone, but it probably acts as a resistant capping layer in the Laughing Whitefish Falls and Chapel Falls exposures as well.

The relatively high percentage of shale in the middle and lower part of the Miner's Castle Member in the Laughing Whitefish Falls section roughly corresponds to the somewhat greater amount of shale found in

TABLE 5.—*Lithology, heavy mineral frequency, and grain size parameters through Chapel Falls section*

Lithology	Thickness in Feet	Percent Frequency by Number					Grain Size Parameters	
		Garnet	Zircon	Leucoxene	Pyrite	Hematite	Mϕ	σϕ
Munising Sandstone								
Miner's Castle Member								
8. Sandstone, white to light yellow, weathers light gray mottled with blue, pink, and yellow, bedding 1 inch to 5 feet, averages 4 feet.	25.0	95	<1	1	1	—	1.23	0.85
		95	1	2	—	1	1.21	0.73
		98	—	1	—	<1	1.31	0.78
		83	—	4	—	13	1.60	0.91
7. Sandstone, medium to deep gray, weathers brown, considerable iron staining, massive bedding.	16.7	42	<1	1	53	—	1.40	0.83
		14	—	1	84	—	1.87	1.23
		50	1	8	35	3	1.78	1.08
6. Sandstone, gray to white, thin shale laminae present, poorly cemented.	0.3	49	—	10	40	—	2.10	1.18
5. Sandstone, medium gray, poorly cemented, single bed.	12.1	48	1	26	16	8	1.45	0.95
4. Sandstone, medium to light green, glauconitic, very poorly cemented.	0.2	33	—	5	27	34	0.93	1.08
3. Sandstone, medium gray, poorly cemented, single bed.	7.8	30	—	20	48	—	2.03	1.45
2. Sandstone and shale, shale is about 10 percent of unit, medium gray, weathers brown, occasional iron staining, cementation variable, bedding 0.5 inches to 10 inches, averages 3 inches.	13.8	42	<1	2	55	—	2.77	1.63
		18	—	8	72	—	2.35	1.53
		25	1	7	65	—	1.93	1.57
Covered interval	3.0							
1. Sandstone and shale, shale is about 10 percent of unit, medium gray becoming white toward top, bedding 0.5 inches to 10 inches, averages 3 inches, cross-bedding common.	11.0	16	<1	21	57	2	1.27	1.63
		16	—	13	66	—	1.40	0.95

the lower and middle part of the Miner's Castle Member in the exposures at Miner's Castle and at Chapel Falls.

SIZE ANALYSIS

Tables 1 through 5 summarize size parameter estimates and heavy mineral analyses of the Mosquito Harbor, Miner's Castle, Laughing Whitefish Falls, Munising Falls, and Chapel Falls sections. The average phi mean diameters of the Jacobsville, Chapel Rock, and Miner's Castle are respectively 1.87, 1.83, and 1.78. The average phi deviation measures are 0.73, 0.71, and 1.07. These averages are calculated from 23 samples of Jacobsville, 13 samples of Chapel Rock, and 60 samples of Miner's Castle.

Grain size variation.—Figure 1 shows the variation of the phi mean diameter in the Miner's Castle Member, as averaged from the four principal sections in which that member is exposed, when it is plotted against the stratigraphic level within the member. Since the Miner's Castle-Chapel Rock contact is not exposed in the Laughing Whitefish Falls, Munising Falls, or Chapel Falls sections it is necessary to use the

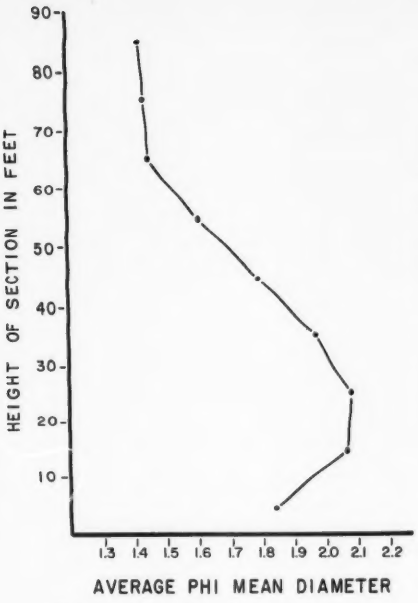

FIG. 1.—Change in phi mean diameter through the Miner's Castle Member.

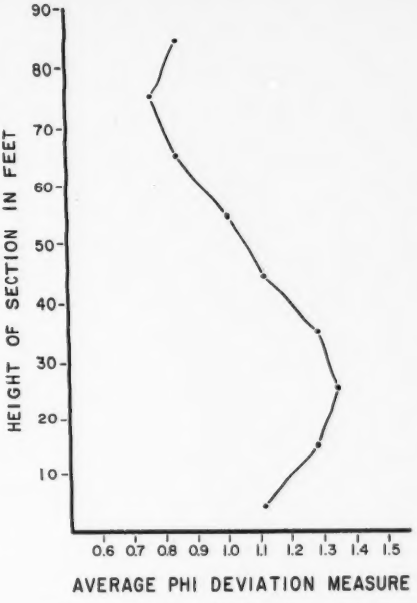

FIG. 2.—Change in phi deviation measure through Miner's Castle Member.

Miner's Castle-Au Train contact as a datum plane. The average phi mean diameter is plotted for each 10-foot interval below this contact. Each parameter estimate used in this plot is appropriately weighted for the thickness of rock which it represents.

The phi mean diameter is greater in the lower middle part of the Miner's Castle Member than it is in either the upper or lower part. This indicates that the grain size is smaller in the lower middle part of the member than it is at either the top or the bottom.

Sorting variation.—Figure 2 shows the variation in the phi deviation measure upward through the Miner's Castle Member. Plotting is by the same method used for Figure 1.

The phi deviation measure, in the four sections analyzed has a greater value in the lower middle part of the Miner's Castle Member than in either the top or bottom. This indicates that the upper and lower parts of the member are better sorted than the lower middle part.

HEAVY MINERAL ANALYSIS

The percent by number of heavy minerals in the Mosquito Harbor, Miner's Castle, Laughing Whitefish Falls, Munising Falls, and Chapel Falls sections is shown in tables 1, 2, 3, 4, and 5 respectively. These tables show only the major constituents of the heavy mineral assemblages. It is apparent, from examination of tables 2 through 5, that a striking increase in percent of garnet occurs from bottom to top of the Miner's Castle Member of the Munising Sandstone. This upward increase is still apparent when authigenic pyrite is not included in the calculations.

Figure 3 shows the mean garnet content of the Miner's Castle Member calculated from all four sections and plotted against stratigraphic level within the member. As in the case of phi mean diameter and phi deviation measure the average for each 10 feet below the Miner's Castle-Au Train contact was calculated and each percentage was weighted according to the stratigraphic thickness which it represented. The dis-

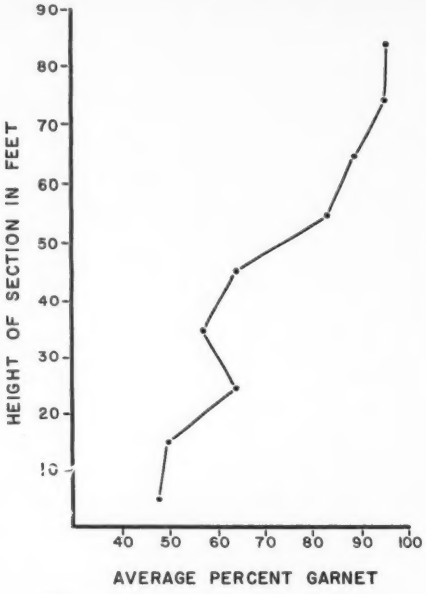

FIG. 3.—Change in heavy mineral garnet content through Miner's Castle Member.

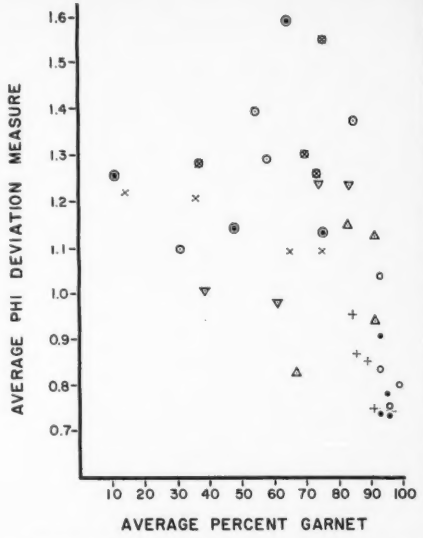

FIG. 4.—Percent garnet-sorting relation in the Miner's Castle Member. Average percent garnet in each 10 foot interval below the Au Train-Miner's Castle contact is plotted against the average phi deviation measure in that 10 foot interval. Similar symbols indicate equal stratigraphic position in the four principal sections plotted.

tinct increase in garnet upward through the member is unmistakable, even though authigenic pyrite was not considered in making the percentage calculations.

It has been shown (fig. 2) that sorting of the Miner's Castle Member consistently changes from base to top. The possibility that the concentration of garnet at any given stratigraphic level is controlled by the sorting at that level has been investigated. In figure 4 the percent of garnet in each 10-foot interval below the Miner's Castle-Au Train contact is plotted against the sorting of that 10-foot interval for each of the four principal sections of the Miner's Castle Member. If garnet content were dependent upon sorting, each set of symbols indicative of equal stratigraphic level on this graph would show a distinct trend. They would show either increasing garnet with decreasing dispersion or decreasing garnet with decreasing dispersion. Neither of these trends is present. Figure 4 therefore demonstrates that garnet concentration is not dependent upon the sorting of the unit from which the sample is obtained.

DISCUSSION AND CONCLUSIONS

The Munising Sandstone may be divided into 3 members. A disconformable relation, probably of local extent, is found between the basal conglomerate and the Chapel Rock Member at Mosquito Harbor. A disconformity between the Chapel Rock Member and the Miner's Castle Member is evident from mudcracks, ripple marks, truncation of cross-bedding and a distinct change in sorting and heavy mineral suites. The disconformity is probably of regional extent.[2]

[2] Howell and Lockman (1939) report an extensive faunal and sedimentational break between the Dresbach and Franconia in Missouri, the Appalachians, the Black Hills, Montana, Nevada, California, and Wisconsin. Concerning Wisconsin, they state: "In Wisconsin the Galesville sandstone is apparently of fresh water origin. The Ironton sandstone of the Franconia stage begins with a basal conglomerate and coarse sandstone; and a marked difference, both in percentage and kind in the heavy mineral content has been found between the Galesville and the Ironton (Wanenmacher, Twenhofel, and Raash, 1934, Am. Jour. of Sci., 5 Ser. Vol. 28, pp. 1–30, p. 11)."

TABLE 6.—*Comparison of the heavy mineral suites of the Upper Cambrian sandstones of Wisconsin and the sandstones of the southern shore of Lake Superior*[1]

	Madison (Wisc.)	Jordan (Wisc.)	Franconia (Wisc.)	Galesville (Wisc.)	Eau Claire (Wisc.)	Mt. Simon (Wisc.)	Jacobsville (N. Mich.)	Munising	
								Chapel Rock Member (N. Mich.)	Miner's Castle Member (N. Mich.)
Garnet	2	75–100	80	0–10	22	2	10	3	52
Zircon	64	10–25	4	25–50	29	18	4	12	1
Leucoxene	11	X	X	1– 5	11	21	29	29	13
Ilmenite	X	X	X	5–10	30	50	12	10	2
Pyrite							4	24	26
Tourmaline	17	5– 10	15	10–25	4	10	2	4	2
Hematite							14	4	2
Magnetite							24	12	X
Apatite			X			X	X	X	X
Rutile	2		X		1	X	X	X	X
Ceylonite				X					
Staurolite					X	X			

[1] After Tyler (1936) in part.

The heavy mineral suite of the Chapel Rock Member is characterized by a high zircon-low garnet content; that of the Miner's Castle Member by a low zircon-high garnet content. Sorting in the Chapel Rock Member is better than that in the Miner's Castle Member.

Chapel Rock Member

The Chapel Rock Member can be correlated with the Dresbach Sandstone of Wisconsin on the basis of stratigraphic position and heavy mineral similarities (table 6). Dissimilarities in heavy mineral percentages, particularly in tourmaline and zircon, between the Galesville (uppermost Dresbach) of Wisconsin and the Chapel Rock Member of the area of study are noteworthy. A heavy mineral suite similar to that of the Galesville does not appear to be present in the area of study. This, in addition to the disconformity between the Chapel Rock and Miner's Castle Members of the Munising and the adduced fresh water origin of the Galesville in Wisconsin suggests that a Galesville equivalent may not be present in the sandstones bordering the southern shore of Lake Superior.

Miner's Castle Member

Oetking (1951) and Hamblin (1958) correlate the Miner's Castle Member of the Munising Sandstone with the Franconia of Wisconsin on the basis of similar heavy mineral assemblages, stratigraphic position, and the occurrence of the middle Franconia *Prosaukia-Ptychaspis* zone in outcrops and drill holes somewhat to the south of the area under consideration. Hamblin (1958, p. 114) recognized the *Prosaukia* zone "in the north side of the Pewabic mine at Iron Mountain and again in a drill-hole core from sec. 7, T. 39 N., R. 26 W." The *Prosaukia* zone has not been found in the sandstones bordering Lake Superior.

The writer proposes that the Miner's Castle Member of the Munising Sandstone was deposited during the transition from a transgressive to a regressive phase of sedimentation (fig. 5). Commonly a transgressive-regressive sequence of sediments is described from basinward exposures in which a sandstone-shale-limestone-shale-sandstone sequence is developed. Such a sequence in the Croixan sediments of southwestern Wisconsin (fig. 5) has been described by Nelson (1956). Nelson did not, however, conclude that the Franconia and Jordan were in any way related. Evidence that the Miner's Castle Member is transitional between the Franconia transgressive sea and the Jordan regressive sea is found in mechanical and heavy mineral analyses.

Grain size variation.—As in more basinward exposures of a transgressive-regressive sequence of beds, it is to be expected that

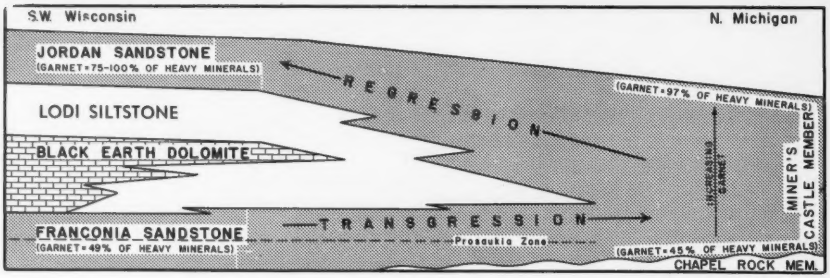

FIG. 5.—Proposed relation of Franconia, Miner's Castle Member, and Jordan Sandstone prior to post-Cambrian erosion. Not to scale. After Nelson (1956) in part.

the grain size of the near shore transitional sand will show coarser size grades at the bottom and top of the section and finer grades in the middle. A graph of the mean grain diameter plotted against the height in the section should exhibit a distinctly finer grade of sand in the middle and coarser grades at both the bottom and top of the section. This is found in the Miner's Castle Member (fig. 1).

Sorting variation.—The bottom and top of the near shore transitional sand sequence represent sedimentary environments which were, at the time of deposition, directly adjacent to the shore. In such an environment current and wave action would be stronger than in environments somewhat farther removed from the shore, that is, in those represented by the middle part of the sand sequence. Winnowing-out and removal of the finer sedimentary grades would be better accomplished in the upper and lower part of such a sequence. Such winnowing-out, associated with constant reworking of the upper and lower parts of the sand sequence by the stronger wave and current action, would lead to markedly better sorting than that exhibited by the middle part. The Miner's Castle Member shows distinctly better sorting at the bottom and top of the member than it does in the lower middle part (fig. 2).

Heavy minerals.—Heavy mineral assemblages are often characteristic of lithologic units. The nature of the source area, the environment of the source area, the transportational, depositional, and post-depositional environments all contribute to the nature of a given heavy mineral suite. Over any appreciable length of time it is to be expected that one or all of these may change, thus leading to a change in the heavy mineral suite vertically through the stratigraphic section. If any marked difference exists between the heavy mineral suite of a transgressive sand and that of its complementary regressive sand it should be reflected by a gradual change in the heavy mineral suite of the transitional near shore sand. It should be noted that if only spot samples of the near shore transitional beds are analyzed and no vertical control is maintained, the heavy mineral suite may show affinities with either the transgressive or regressive sands in more basinward exposures, or possibly some affinities with both Unless sampling and analysis are carried out carefully throughout the section the true transitional nature of the beds could easily be overlooked. Figure 3 shows a marked increase in garnet from approximately 45 percent of the heavy minerals at the base of the Miner's Castle Member to almost 100 percent at the top. This increase upward is striking in all sections studied. The change is from a heavy mineral assemblage which is similar to that of the Franconia Sandstone of Wisconsin[3] to one which

[3] Tyler (1936) shows the heavy mineral suite of the Franconia sandstone of Wisconsin to be 80 percent garnet (table 6). However, Pentland (1931) from a thorough study of the heavy minerals of the Franconia and Mazomanie sandstones of Wisconsin, concluded that the Mazomanie should be correlated with the upper part of the Franconia. The average percentage of garnet in the heavies of the two formations calculated from 115 of Pentland's analyses is 49 percent.

is similar to that of the Jordan Sandstone (table 6).

Changes in the mean grain diameter and in dispersion in the upper part of the Miner's Castle Member indicate a regressive phase of sedimentation. Berg (1954 p. 887) states, in reference to the Franconia sediments of southwestern Wisconsin, that "There is no evidence for regressional sedimentation." Heavy mineral analyses in the present study indicates that the transgressive Franconia Sandstone grades upward through the transitional Miner's Castle into the regressive Jordan Sandstone.

This relationship in no way conflicts with the occurrence of the *Prosaukia* zone in Franconia Sandstone to the south of the area of study (Stumm, 1956; Hamblin, 1958, p. 114). The smallest mean grain diameters and the poorest sorting in the Miner's Castle Member occur in the lower middle part of the member (figs. 1 and 2). This indicates that the transgressive phase of the Franconia-Jordan sea was notably more rapid than the regressive phase. That the lower part of the Miner's Castle Member exposed along the Lake Superior shore is a time equivalent of the *Prosaukia* zone of Wisconsin is a distinct possibility (fig. 5).

ACKNOWLEDGMENTS

The writer is indebted to Dr. A. L. Lugn and Mr. Walter Sadlick, of the University of Nebraska. Dr. Helen Martin of the Michigan Geological Survey visited the writer in the field and provided helpful suggestions. Dr. Louis I. Briggs of the University of Michigan read and criticized the manuscript.

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JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 29, NO. 1, PP. 16–29
FIGS. 1–6, MARCH, 1959

GEOLOGY AND PALEONTOLOGY OF NORWEGIAN SEA BOTTOM CORES[1]

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ABSTRACT

Nine sediment cores from the southeastern part of the Norwegian Sea, collected from depths between about 700 m and 3000 m, have been examined. A gray, unsorted, sandy and pebbly clay, assumed to represent a glacial-marine sediment, is found to cover the surface of the sea bottom down to about 900 m depth, and to continue underneath more recent sediments at greater depths. These latter include a 10–15 cm thick top-layer of foraminiferal marl, with a $CaCO_3$ content reaching 48 percent.

An examination of the Foraminifera distribution of a core collected from a depth of about 3000 m was carried out. The gray glacial clay is poor in Foraminifera, and the major part of the fauna consists of benthonic forms which live in lesser depths than where they are found, and therefore must be regarded as displaced. As this displaced fauna is restricted to the glacial-marine sediments of the core, it is assumed that it is transported to deep water by drifting ice. The low percentage of planktonic Foraminifera is thought to indicate rapid sedimentation of clastic material. The upper part of the glacial clay, which shows an increase in fineness upwards, also shows a marked decrease of the displaced benthonic fauna and a corresponding increase in the planktonic population. This is thought to indicate an amelioration of climate, with less ice-drift transportation, but still with rapid sedimentation of clastic material. The great increase in the Foraminifera population in the upper 30 cm of the core, especially of planktonic forms, indicates further amelioration of climate with clastic sedimentation having been of less importance.

INTRODUCTION

Our knowledge of the bottom deposits of the southern part of the Norwegian Sea is derived mainly from the work of the Norwegian North-Atlantic Expedition 1876–1878 (Schmelck, 1882), and of the Danish Ingolf-Expedition 1895–1896 (Böggild, 1899). For the northern areas of the Norwegian Sea our main source of knowledge is again primarily the Norwegian North-Atlantic Expedition. In addition the "Belgica" Expedition in 1905 (Böggild, 1907) studied bottom deposits from the sea areas off East Greenland and over to Spitzbergen, while off the northwest coast of Spitzbergen samples were collected by the "Nautilus" Expedition in 1931 (Stetson, 1933).

Descriptions of sediments from the Faerö-Shetland ridge, and from the sea area north and west of northern Scotland, are provided in the reports of the "Michael Sars" North Atlantic Deep Sea Expedition, 1910 (Chumley, 1910, Murray and Hjort, 1912, Peach, 1912). More recently the present author has studied the sediments of the continental terrace in an area off the Möre-Romsdal coast, and has also made a study of a series of samples collected between Iceland and Norway (Holtedahl, 1950, 1955).

From the work of the various expeditions, especially the Norwegian North-Atlantic Expedition, the main character and distribution of the bottom deposits of the Norwegian Sea area are known fairly well. According to these observations, gray clay is widely distributed over the sea bottom, from the shallowest coastal regions down to the greatest depths. Downwards from a depth of 1500 m–2000 m, however, this gray clay is covered by a brown sediment, distinguished, apart from its color, by its fairly high content of Foraminifera of types which do not occur in the more elevated parts of the sea bottom, where the surface consists of gray clay. This brown sediment is termed by Schmelck *Biloculina* clay, and by Böggild and Murray *Globigerina* clay. Between depths of about 900 m and 1500–2000 m the gray clay is, according to Schmelck, covered by a gray-brown clay, rather poor in Foraminifera, and termed by him transition clay.

The admixture of sand and pebbles, very frequently observed in the gray clay, was by

[1] Manuscript received May 28, 1958

Schmelck explained as material dropped from ice-bergs, and the clay itself as fine glacial detritus originated from the melting glaciers on land during the latter part of the Pleistocene.

RECENT STUDIES OF CORE SAMPLES COLLECTED IN THE SOUTHEASTERN NORWEGIAN SEA

During various cruises, carried out by the Bergen University vessel "Armauer Hansen" and the Fishery Research vessel "G. O. Sars" from 1950 and onwards, core samples and dredge samples have been collected by the writer from areas off the Norwegian west coast and in the southeastern part of the Norwegian Sea. In figure 1 are shown the location and the character of a series of core samples from the continental slope and the Norwegian Sea basin. Samples 6, 7, 8 and 9 were collected with a lined gravity corer, built on the same principles as the free-fall coring tube described by Hvorslev and Stetson (1946), and with a lead weight of about 100 kg, the others with a smaller corer without liners.

It will be seen that gray clay is present at the surface of the sea bottom down to a depth of about 1000 m. At 1500 m depth the gray clay is covered by a thin veneer of

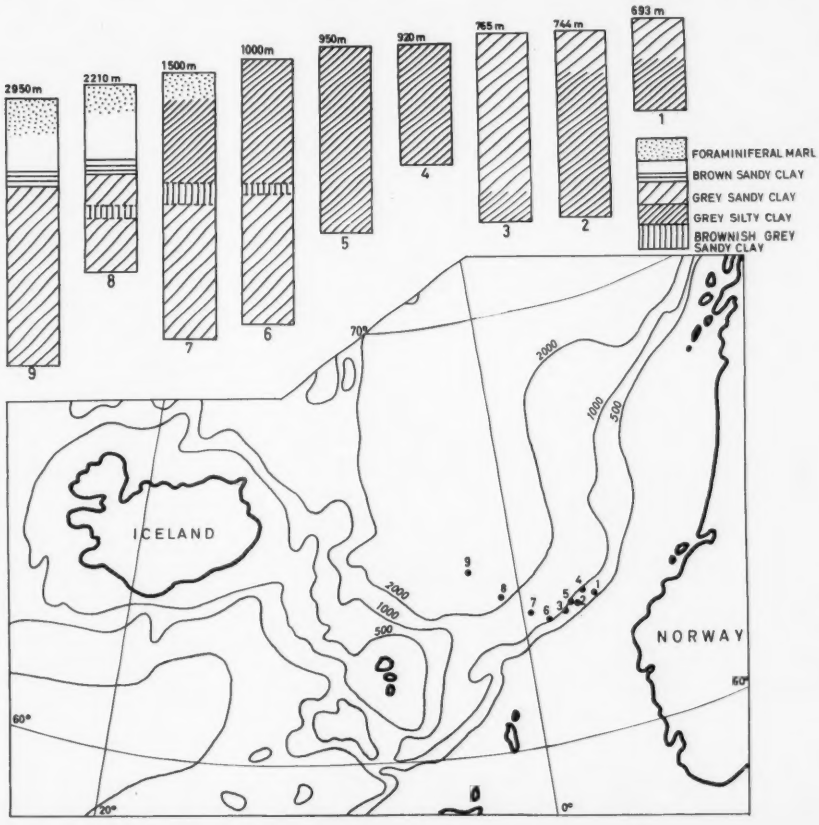

FIG. 1.—Location and character of a series of core-samples from the southeastern Norwegian Sea.

foraminiferal marl, which is also found as a surface layer at greater depths. At a depth of about 2000 m, however, a brown sandy clay, with a mechanical composition not very different from the gray clay, is found between the foraminiferal marl and the gray clay. This brown deposit probably corresponds to Schmelck's transition clay.

Gray Clay

The gray clays vary somewhat in mechanical composition in the various samples and also in the different zones of the same core. They are sandy or silty clays, to some extent sandy silts, with or without pebbles, and the sorting is generally poor. Stratification is normally lacking.

The cores taken from the upper part of the continental slope, between a depth of 700 m and 950 m, vary considerably. At 693 m the surface is covered by a pebbly sandy clay with poor sorting, while underneath this, the sediment is a well sorted silty clay. At 744 m depth the core consists of sandy clay, but with a larger sand content in the top part than in the bottom part. The sediment contains no pebbles and the sorting is poor. Lower down the slope, at 765 m and 920 m, pebbles are present all through the cores right up to the surface, but the two sediments are very different; the core taken at 765 m is a coarse, sandy silt, while the other taken at the greater depth is a silty clay with a small sand content. At 950 m depth the sediment is a clayey silt, with a small admixture of sand, but with no grains of pebble size.

The textural character of the cores taken at depths between 1000 m and about 3000 m is shown in figures 2 and 3. With regard to the gray clay a marked difference in texture is seen in the lower and upper parts of the cores. This is especially well marked in cores 6 and 7, taken at depths of 1000 m and

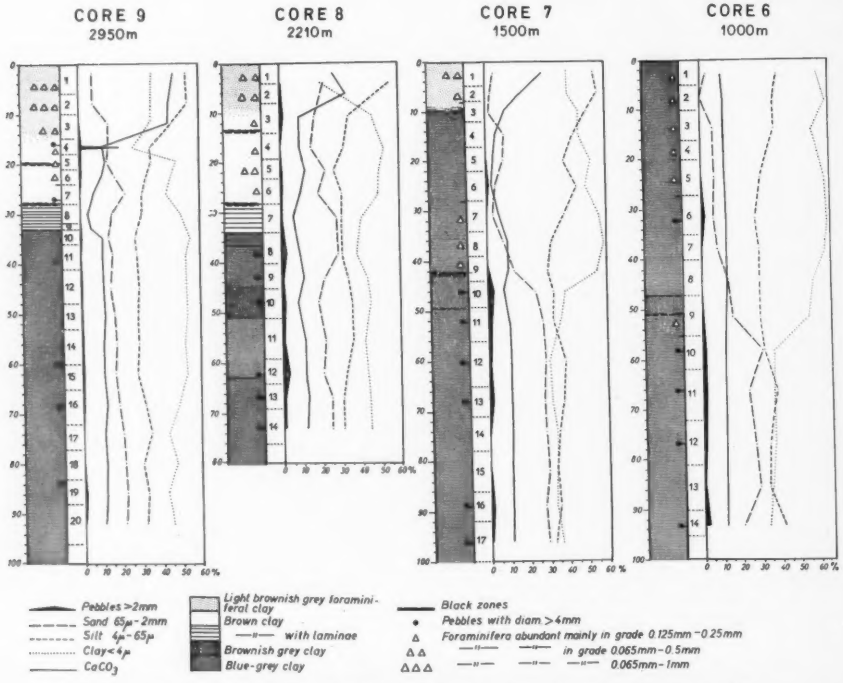

FIG. 2.—Size distribution and other characters of cores from the Norwegian Sea. Depths in cores in cm.

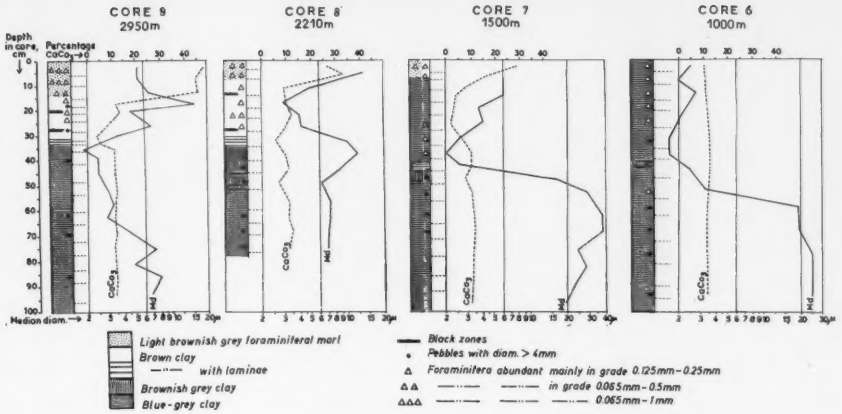

FIG. 3.—Median diameters and $CaCO_3$ content of cores from the Norwegian Sea.

1500 m respectively, and is conspicuously demonstrated by the median curves.

The lower parts of cores 6 and 7 consist of clay, silt, and sand in much the same proportions, and with an admixture of pebbles. The upper parts, however, have a much higher clay content and correspondingly much lower sand content, while the silt values vary but little. Pebbles also occur in this upper sediment. Another difference between the lower and upper clays is shown by their content of Foraminifera. While tests of Foraminifera are very scarce in the lower sections, they appear fairly abundantly in the upper parts.

The gray clays of cores 8 and 9 do not exhibit the same very marked change of textural character in their lower and upper parts as do cores 6 and 7. In core 9 there is a more gradual increase in fineness upwards, but in core 8 the increase in fineness is first apparent in the brown clay above. The composition of the gray clay in these latter cores is fairly similar to the clay of the lower parts of cores 6 and 7, consisting of clay, silt, sand, and pebbles, but with a somewhat higher clay content.

Brown Clay

In the core samples 8 and 9 there is a very sharp boundary between the gray clay and a brown clay above. The boundary is marked by a thin dark-brown zone. Above the boundary the sediment shows a number of thin laminae, rusty brown in color and less than 1 mm in thickness, separated by thicker zones of a grayish-brown color. Above the laminae the brown clay does not show any stratification, except for a few fine, dark layers. Higher up, the brown clay gradually changes into a light brownish-gray foraminiferal marl which forms a 10 to 15 cm thick top layer.

The laminated zone of the two cores does not show any marked difference in grain size distribution from the sediment below. In core 9 there is a slight increase in coarseness, but this is even more noticeable in the zone above. Probably the greatest difference is shown by the marked decrease in $CaCO_3$ content, especially well marked in core 9, and by its scarcity of microfossils.

In the brown zone above, core 8 shows some increase in fineness, while core 9 shows some increase in coarseness, but both sediments are still silty clays with a considerable sand content of inorganic origin, and with a small admixture of pebbles. Tests of Foraminifera are, however, much more abundant than in the zones below.

Foraminiferal Marl

A foraminiferal marl is present as a top layer of the cores 7, 8 and 9, and has a slightly greater thickness in the sample taken at the greatest depth than in the more

shallow ones. The change from brown clay to foraminiferal marl is quite gradual, but the difference between this latter sediment and the others is very pronounced, and is due mainly to its large content of material of organic origin. The sand and silt fractions, shown by the size distribution curves, consist mostly of tests of Foraminifera, and the great amount of organic material present is shown by the high $CaCO_3$ values.

Brownish-Gray Clay

In figures 2 and 3 are shown a 3–5 cm thick zone of a brownish-gray clay which is noticeable in the cores 6, 7 and 8. There is nothing in its mechanical composition which distinguishes it from the gray clay below and above, but, apart from its color, it has a slightly lower $CaCO_3$ value, which may be of some significance.

From the description above, it seems clear that the gray clay of cores 8 and 9, and of the lower parts of cores 6 and 7, is a complex type of deposit which is very similar to the "glacial marine sediments" described by Philippi (1912) and others from the region of the Antarctic ice-front and elsewhere. It can be regarded as a glacial sediment, where part of the material has been carried to the depositional site by drifting ice. It is also apparent that the two cores procured at the greatest depths and also at the greatest distance from land are finer than the two cores from lesser depths.

The foraminiferal content is very low, that is less than one percent, and the $CaCO_3$ content fairly constant, about 11–12 percent, mainly present in the silt and clay grades. In attempting to make a correlation between the different zones of the cores, it seems natural to assume that the lower, coarse part of the gray clay in cores 6 and 7 and the gray clay below the brown layer in cores 8 and 9 belong to the same stratigraphic unit. It is also likely that this deposit can be correlated with the pebbly and sandy silts and clays encountered on the bottom surface higher up on the continental slope, between depths of about 600 and 900 m. One must assume that the glacial marine sediments were deposited both in shallow coastal water and in the deeper parts of the sea during a time when drifting ice, carrying clastic material, was abundant in the sea. Currents were, however, especially effective in washing out the fine material of the sediments down to present depths of about 600 m.

The sudden change in texture, with an increased fineness of the sediments upwards in the core, seen especially clearly in cores 6 and 7, must be due to very marked climatic changes, which would result in increased melting of the glaciers and thereby in an increased amount of fine clastic material being transported into the sea. This amelioration of climate has probably also resulted in a temporary disappearance of drifting ice from the sea areas, as is suggested by a zone in the cores which is lacking in coarse material. The fairly common appearance of Foraminifera in a more or less extensive zone may also suggest a change in environmental conditions, probably a change to slower deposition due to climatic changes.

In core 8 the change in texture upwards in the core, noticable in the brown clay, is not so striking as that observed in the upper parts of the gray clay in cores 6 and 7, but there is a definite increase in fineness, a definite increase in the Foraminifera content, and a zone devoid of coarse material, all of which would seem to warrant a correlation with the upper gray clays of cores 6 and 7. If this is justified, it would mean that the same zone is gray in color above a depth of about 1500 m, and brown in color below this depth. According to Schmelck (1882) the difference in color between the brown clay and gray clay is merely due to the state of oxidation.

Correlation with core 9 is more difficult: here the maximum fineness of the sediment is found just below the brown deposit, where Foraminifera are scarce. Higher up in the core, where Foraminifera are common, the sediment becomes coarser. It is probably most natural to correlate the sharp boundaries between the gray and brown deposits of cores 8 and 9. In spite of considerable difference in texture, the zones above the boundaries show certain points of resemblance, in that both are laminated and show a drop in carbonate content.

The slight increase in the coarseness of the sediments near the surface of the cores, in conjunction with a very marked increase in

the content of Foraminifera and a rise in the calcium carbonate content, indicates a decreasing amount of clastic material being transported to the sea, and probably also an increased production of Foraminifera. The lack of pebbles and coarse sand shows that drifting ice was absent, and the small amount of clastic material carried to the sea indicates that the contribution of glacial meltwater was slight.

FORAMINIFERA DISTRIBUTION IN CORE 9 (DEPTH: 2950 M)

The approximate percentages of Foraminifera in the various size grades of the different zones of core 9 are shown in table 1 and in figure 4.

It will be seen that Foraminifera are very scarce in the gray clay and in the lower part of the brown clay, but increase in amount, and also in size, from the middle zone of the brown clay upwards.

The variation in the $CaCO_3$ content of the total samples, as well as the $CaCO_3$ content of the various size grades of two representative samples, are shown in table 2.

It is seen that the large and sudden increase in $CaCO_3$ appears in sample 3, only about 15 cm from the surface of the core. The increase in Foraminifera content which begins in sample 6 does not show up in the $CaCO_3$ values. The $CaCO_3$ analyses of various size grades show that in the foraminiferal marl the largest grain sizes have the highest $CaCO_3$ values, while the glacial marine clay has the highest $CaCO_3$ values in the finest grades. This probably means that in the glacial clay the $CaCO_3$ is mostly present as

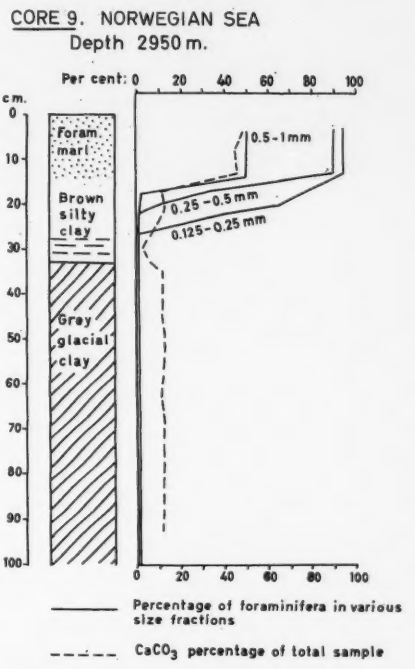

FIG. 4.—Foraminifera content of core 9.

TABLE 1.—*Approximate percentages of Foraminifera in various size grades*

	Samples																
Size fraction	1	2	3	4	5	6	7	8	10	11	12	13	14	15	17	19	20
0.5–1 mm	50	50	50	X	X												
0.25–0.5 mm	90	90	90	33	12	X	X	X	X	X	X	X	X				
0.125–0.25 mm	95	95	95	75	65	40	X	X	X	X	X	X	X	X	X	X	X

X < 1 percent.

TABLE 2.—*Percentages of $CaCO_3$ in total sample and in various size grades*

Sample	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
Total	48.3	45.3	45.6	11.9	13.1	11.2	6.8	3.7	5.7	11.0	11.7	11.7	12.3	11.6	11.5	12.5	11.4	11.8	11.9	11.8
0.125–0.25 mm			79.7																3.2	
0.065–0.125 mm			54.4																3.2	
0.065 mm			29.4																15.3	

TABLE 3.—*Distribution of Foraminifera in core 9, Norwegian Sea, N. 64°15′, W. 2°35′, depth: 2950 m.*

Sample	1	2	3	4	5	6	7	8	10	11	12	13	14	15	17	20
Cm from top of core	0–6	6–10	10–15	15–18	18–21	21–24	24–28	28–32	33–36	36–41	41–48	48–53	53–60	60–65	72–77	88–96
Planktonic population																
Globigerina bulloides																
Globigerina eggeri																
Globigerina pachyderma	VA	VA	VA	VA	VA	VA	VA	VA	VA	A	C	C	C	C	R	R
Globigerina quinqueloba																
Globorotalia truncatulinoides									R				R			
Orbulina universa		R	R													
Benthonic population																
Angulogerina angulosa									R							R
Bolivina sp.																R
Buccella sp.							R					R				
Bulimina aff. marginata						R	R		R	R	C	C	R	R	R	R
Cassidulina norcrossi																
Cassidulina islandica				R		R				C	C	C	C	C	R	C
Cibicides aff. lobatulus									R	R	R	R		R		
Cibicides wüllerstorfi	R	R	R	R	R	R	R									
Elphidium bartletti																
Elphidium incertum					R	R	R	R	A	A	VA	VA	VA	VA	VA	VA
Elphidium orbiculare																
Elphidium subarcticum																
Epistominella exigua	R	R	R	R	R	R	R									
Eponides umbonatus	R	R	R	C	R	R	R		R							
Gyroidina lamarckiana		R	R													
Lagenidae	R	R	R													
Miliolidae																R
Nonion barleanum			R			R								R		R
Nonion labradoricum										R			R			R
Parafissurina sp.	R	R	R													
Pullenia bulloides											R	R				
Pyrgo aff. murrhina	R	R	R	R	R								R			
Uvigerina sp.						R										
Virgulina sp.															R	

R: 1–5 percent of population (Rare).
C: 5–25 percent of population (Common).
A: 25–50 percent of population (Abundant).
VA: 50–100 percent of population (Very abundant)

very fine, crushed shell-debris, and only to a small extent as unbroken tests.

In table 3 are listed the identified Foraminifera of core 9 and their distribution and abundance in the various zones of the core.

Planktonic Population

Globigerina pachyderma (Ehrenberg) is the most common species, and nearly 98 percent have sinistral coiling throughout the core. It is a cold water form, at present living in arctic and subarctic water, and described from the Polar Sea as well as from the northern North Atlantic. *Globigerina bulloides* d'Orbigny is a more temperate form, but may be adapted to rather cold water, and is quite abundant in the Polar Sea (Kiær, 1899).

The Globigerina-tests are very scarce in the lower and middle parts of the gray glacial clay, but show a marked increase in number in the upper part, and a maximum in the brown clay and foraminiferal marl. The presence of cold water planktonic foraminifera in all sections of the core does not give any indication of large-scaled climatic changes. The occurrence of *Orbulina universa* d'Orbigny in the upper parts of the core may, however, be an indication of warmer sea temperatures. Brady gives a range of this species from Novaya Zemblya to 50° S. Latitude.

Benthonic Population

In the major part of the gray glacial clay the benthonic foraminiferal fauna constitutes about 80–90 percent of the total Foraminifera population, while in the brown clay and foraminiferal marl it is only 10 percent or less. It is possible to distinguish between various faunal groups which are characteristic of different parts of the core.

1. The gray glacial clay is characterized first and foremost by the genus *Elphidium*. *Elphidium incertum* (Williamson) and *E. subarcticum* Cushman are particularily common and the total *Elphidium* fauna occurs in an abundance of between 60 and 80 percent of the Foraminifera population in the lower and middle part of the gray clay (fig. 5). In the upper part it decreases in amount, and is completely absent in the laminated, brown clay zone higher up. A few specimens are present in the sample above the laminated zone, but are completely lacking in the upper brown clay and in the foraminiferal marl.

Apart from *Elphidium spp.*, *Cassidulina norcrossi* Cushman, *C. islandica* Nörvang, *Bulimina aff. marginata* d'Orbigny and *Cibicides aff. lobatulus* (Walker and Jacob) are the most common species in the gray glacial clay. *Angulogerina angulosa* (Williamson), *Nonion labradoricum* (Dawson) and *Pullenia bulloides* (d'Orbigny) are also present.

Elphidium incertum (Williamson) is widely distributed in the Arctic and in the North Atlantic today. It is common along the Icelandic and Norwegian coast, and is reported from the coast of Maryland to the Arctic in the western Atlantic. It is mostly regarded as a shallow water and near-shore form, yet Phleger describes *E. incertum* from deep water along the continental slope in the Cape Henry-Cape Cod area (Phleger, 1942). Cushman and Henbest (1940) report *E. incertum* and variations from their North Atlantic cores, and suggest their present distribution in deep water as due to ice-transport.

Elphidium subarcticum Cushman is common in the western North Atlantic and is reported from Arctic as well as Antarctic waters (Cushman, 1920, Heron-Allen, 1932). It may be classified as a cool-temperate form.

Cassidulina norcrossi Cushman is reported from areas off Greenland and off Iceland and in the Arctic, as well as from the continential shelf of the western Atlantic, where it reaches its greatest frequency at depths of less than 300 m.

Cassidulina islandica Nörvang, var. *minuta* Nörvang is reported from Arctic and Antarctic waters. It is found off Iceland and off the coast of Norway and west-coast of Sweden, as well as along the east-coast of United States.

Bulimina marginata d'Orbigny has a present distribution mainly in temperate waters (Nörvang, 1945). Although it has been found at all depths, its greatest percentage frequencies seem to be in water shallower than about 700 m. Goës (1894) describes it from off Norway and Spitzbergen at depths between 100 and 700 m, and Phleger (1942) from the western North

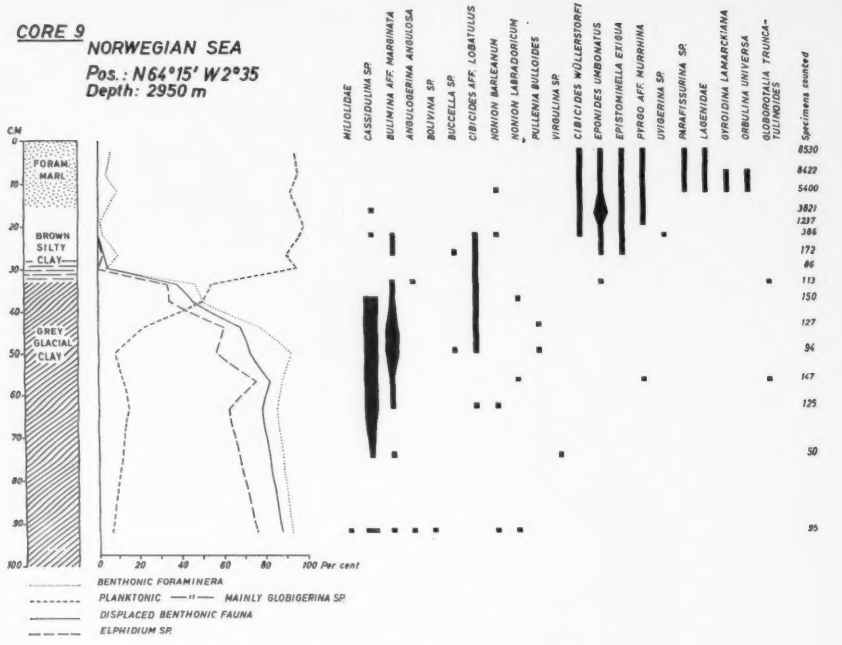

FIG. 5.—Foraminifera distribution of core 9.

Atlantic, mainly in water shallower than about 700 m.

Cibicides lobatulus (Walker and Jacob) is a very widespread attached form, widely reported in the North Atlantic as well as in Arctic waters. It is probably characteristic of shallow water, and is in deeper water often associated with displaced faunal assemblages.

Angulogerina angulosa (Williamson) is widespread in the Atlantic, and is abundant off the northeast coast of the United States north of Cape Hatteras. It is also reported from off Greenland and in the Arctic. From Skagerak it is described by Höglund (1947) from depths between 100 m and 700 m, but with maximum frequences at 200–250 m depth.

Nonion labradoricum (Dawson) is widespread in the western North Atlantic and ranges northwards from Cape Cod to the Arctic. Its main distribution is the arctic parts of the North Atlantic.

As will be seen, the foraminiferal fauna of the gray glacial clay corresponds to a fauna which at the present time to a great extent occurs in the Arctic and in the North Atlantic region. Furthermore, a great part of the benthonic fauna of the gray clay corresponds to a present day fauna which does not live at the great depth at which it is found in the core, and must be regarded as displaced.

2. The brown clay has a benthonic fauna which is distinctly different from that of the gray clay. The lower part shows the first appearance of *Cibicides wüllerstorfi* (Schwager), *Epistominella exigua* (Brady) and *Eponides umbonatus* (Reuss). These forms increase in number upwards in the core and show a great abundance in the upper part of the brown clay.

Cibicides wüllerstorfi has a widespread occurrence and is common in the Atlantic and in the Arctic Ocean. In the North Atlantic it does not usually occur above a

depth of 900 m. *Epistominella exigua* has also a widespread occurrence and is usually found at depths exceeding 275 m. *Eponides umbonatus* is found commonly in cores and surface samples, from water deeper than 42 m in the North Atlantic.

3. The foraminiferal marl has faunal characters very similar to those of the upper brown clay except for the appearance of abundant Biloculines, especially *Pyrgo* aff. *murrhina* (Schwager). This form may attain a very large size exceeding 1 mm in diameter. *Pyrgo murrhina* is a species widely distributed in the North Atlantic at depths greater than 200 m.

INTERPRETATION OF RESULTS

The very small content of Foraminifera in the gray glacial clay compared to the great abundance characteristic of the middle and upper part of the brown silty clay, and of the foraminiferal marl, must be due to great depositional differences. During the time when the gray glacial clay accumulated, sedimentation of clastic material must have been comparatively rapid. Fine glacial detritus from the melting glaciers was carried in abundance to the deeper parts of the sea, and drifting ice dropped its load of very heterogenous material. As the major part of the small Foraminifera content of the glacial clay consists of benthonic forms which must be regarded as displaced, it has probably been incorporated in the ice in shallow water and subsequently transported to deeper water by drifting ice. The fact that the $CaCO_3$ content of the glacial clay is higher in its finer fractions than in its coarser, and that this $CaCO_3$ is of organic origin, seems to support this assumption of ice transport. Only a very small fraction of the Foraminifera found in the glacial clay can be considered autochthonous, and of this fauna the major part consists of planktonic forms.

In figure 5 is shown how the proportions between planktonic and benthonic Foraminifera change in the upper part of the gray glacial clay. There is a very marked increase in the planktonic population and a corresponding decrease in the number of benthonic forms. Looking at the displaced Foraminifera population it will be seen that it shows a definite, but gradual decrease in the upper half of the gray clay. The proportion between displaced and autochthonous benthonic forms does not, however, alter very much as will be seen from figure 6, and the benthonic population is mainly displaced throughout the gray clay.

The relative increase in the planktonic fauna in the upper part of the gray clay is probably caused by a decreased sedimentation of ice-transported material. The total foraminiferal population is still very small. This is in agreement with the size frequency distribution of the gray clay, which shows an increasing fineness upwards towards the brown clay (fig. 3). It is also in agreement with the conclusion drawn from the study of the mechanical composition of the cores 6 and 7, where a very marked increase in fineness was noted in the middle parts of the cores, which pointed to a climatic change from a cold climate with an abundance of drifting ice in the sea to a milder climate with increased melting and decreased ice-transportation.

In the laminated zone at the bottom of the brown, silty clay, the foraminiferal content is still very low and the major part of the fauna is planktonic. The few benthonic forms present are all displaced. The distinct boundary between the gray clay and the brown clay above, the lamination of the zone at the bottom of the brown clay and the low $CaCO_3$ content of this zone compared to the zones above and below, are facts which may suggest deposition by turbidity currents. The very small content of displaced shallow water Foraminifera does not, however, suggest that currents came from shallow water. Also the sharp boundary between the gray and brown clay is a feature which is characteristic of large parts of the Norwegian Sea, and does not seem to be a phenomenon caused by local turbidity currents. The question is whether this sudden change in the state of oxidation of iron components of the sediments may not have some connection with a sudden change in the hydrographic conditions in the Norwegian Sea, which again may be related to the change of sill-depth of the Iceland-Faeroe ridge.

The brown clay above the laminated zone shows a very abrupt increase in planktonic Foraminifera and at the same time a continued decrease in displaced benthonic

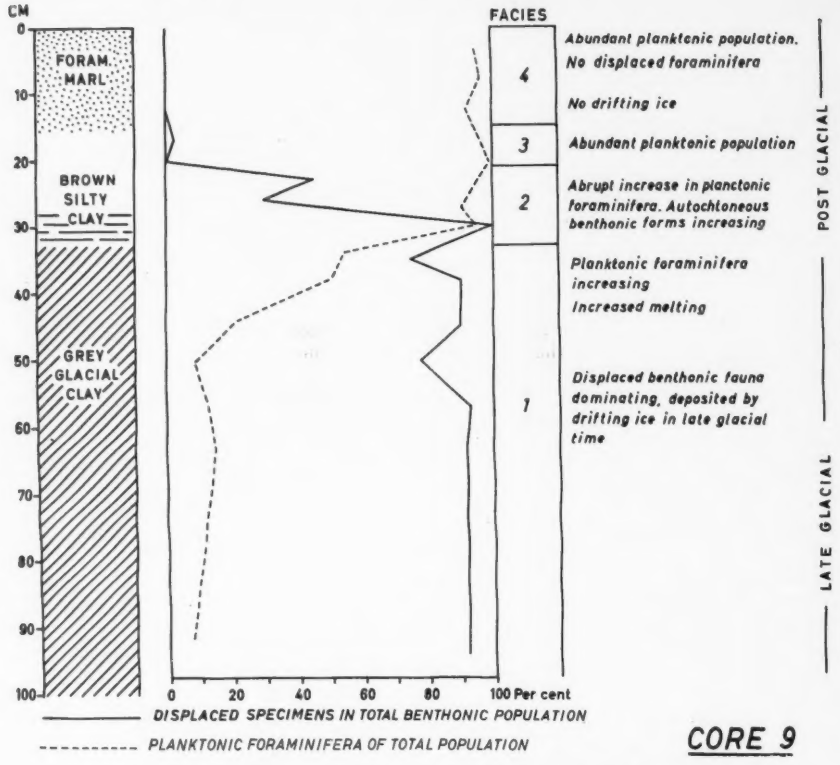
CM
FACIES
FORAM. MARL
BROWN SILTY CLAY
GREY GLACIAL CLAY
Abundant planktonic population. No displaced foraminifera
No drifting ice
Abundant planktonic population
Abrupt increase in planctonic foraminifera. Autochtoneous benthonic forms increasing
Planktonic foraminifera increasing
Increased melting
Displaced benthonic fauna dominating, deposited by drifting ice in late glacial time
POST GLACIAL
LATE GLACIAL
0 20 40 60 80 100 Per cent
DISPLACED SPECIMENS IN TOTAL BENTHONIC POPULATION
PLANKTONIC FORAMINIFERA OF TOTAL POPULATION
CORE 9

FIG. 6.—Distribution of displaced foraminifera in total benthonic population of core 9, and assumed development of sedimentation during Late- and Post-Glacial time.

Foraminifera and an appearance of a quite different benthonic fauna consisting to a great extent of *Cibicides wüllerstorfi*, *Eponides umbonatus* and *Epistominella exigua*. This suggests that ice-transport of debris became less and less significant and that living conditions for benthonic Foraminifera at the great depths improved. It also suggests that sedimentation of clastic material took place at a slower rate—or that the populations of planktonic foraminifera became more abundant in the upper layers of the sea—probably both. That some ice transport took place at this time is seen by the presence of pebbles. The pebble-free zone is, however, not quite without displaced Foraminifera, even if their occurrence is very small.

In the lower part of the foraminiferal marl the displaced benthonic fauna is completely absent, and the benthonic Foraminifera are the same forms as in the brown clay below, with the addition, especially, of Biloculines. The planktonic forms, that is Globigerinids, are very abundant, and the medium sand fraction is almost entirely of foraminiferal tests. The lack of displaced Foraminifera and the corresponding lack of pebbles and inorganic sand, with the exception of the finest grades (the coarse inorganic sand present consists of aggregates of fine material forming casts of foraminiferal tests), does show that sedimentation from floating ice did not any longer take place, and also that sedimentation of fine clastic material

was much less important than before. The sediment must have been laid down during periods when the glaciers from the waning ice-sheets did not any longer reach the coastal waters, and when sediments carried by glacial streams were of no significance for the deposits of the deep ocean.

Dating of Events

A correlation of the climatic changes, as suggested by the cores, with the known climatic changes during Late-Glacial and Post-Glacial times, cannot yet be made with any degree of accuracy. It may be of interest, however, to draw a comparison between the Foraminifera fauna of the core from the Norwegian Sea and the fauna known from Late-Glacial and Post-Glacial deposits in Sweden and Norway.

Hessland (1946) and Brotzen (1951) have described a Late-Glacial fauna from Swedish deposits which has *Elphidium incertum, Elphidium incertum* var. *clavatum, Cassidulina crassa* and *Nonion labradoricum* as characteristic forms, with *Elphidium* sp. greatly dominating. *Cibicides lobatulus* becomes fairly common in the later parts of Late-Glacial times. The oldest deposits are poor in species as well as in individuals, but the number of individuals as well as species increase in the younger deposits. In the Post-Glacial deposits the number of species increase still further, particularly of boreal and lusitanian forms, but *Elphidium incertum* and *E. incertum* var. *clavatum* are still very abundant, with the latter in majority. Another very characteristic form is *Streblus beccarii* (Linné) which Munthe has used as a fossil indicator of Post-Glacial deposits. *Bulimina marginata* is also present in the Post-Glacial sediments.

In Norway Feyling-Hanssen (1954, 1955, 1957) has studied the Foraminifera faunas of Late- and Post-Glacial sediments from the Oslofjord area. He finds in the Late-Glacial Yoldia-clay *Elphidium incertum* var. *clavatum, Cassidulina crassa, Nonion labradoricum, Cibicides lobatulus* and *Astronion gallowayi* as the most common forms, with *E. incertum* var. *clavatum* dominating (about 70 percent). In the succeeding Late-Glacial Arca-clay *Elphidium incertum* var. *clavatum* is still the dominating form (about 45 percent), with *Cassidulina crassa, Virgulina* aff. *davisi, Pullenia q. minuta, Cassidulina laevigata* and *Elphidium incertum* as important species.

From a clay deposit, dating from the transitional period between Late-Glacial and Post-Glacial times, a frequent occurrence of various *Elphidium* species was also found to be characteristic.

In the Post-Glacial Isocardia-clay the most characteristic species were *Bulimina marginata* (about 50 percent), *Elphidium incertum, Nonion pompiloides, Cassidulina laevigata, Elphidium i. clavatum, Elphidium i. selseyensis* and *Virgulina fusiformis.*

From this faunistic comparison alone it seems natural to correlate the gray clay of the Norwegian Sea cores with the Late-Glacial deposits of Norway and Sweden. The dominance of *Elphidium* sp. in the oldest Late-Glacial accumulations, described by Hessland, Brotzen and Feyling-Hanssen is very striking, as it is in the major part of the gray clay described from deep water in the Norwegian Sea. The presence of *Elphidium* sp. in the Post-Glacial shallow water deposits from Norway and Sweden, and the lack of these forms in the younger parts of the cores from deep water, is a good indication of the displaced character of the gray clay fauna.

A major question is, at which time the very marked climatic change took place which resulted in decreased ice transport and a change in the foraminiferal fauna from mainly displaced benthonic forms to mainly planktonic forms. Probably this marked climatic change took place in the transition period between Late- and Post-Glacial times. A similar, very sharp change in climate has been recorded in cores from the Atlantic Ocean and the Carribean Sea, and has been dated by C^{14} methods to about 11,000 years (Ericson, Broecker, Kulp, Wollin, 1956), but occurring in a time interval in the order of 1000 years. Oxygen paleotemperature curves, as well as climatic curves based on variations in frequencies of species of Foraminifera, agree in showing that the transition from glacial to post-glacial climate was marked by a pronounced temperature rise of the upper layer of water. It has also been suggested that the rapid deglaciation did not set in until after the Valders readvance.

This would mean that the lower part of the gray glacial clay from the Norwegian Sea was deposited before and during the time of the Younger Tundra Phase (Ra, Mid-Swedish moraines, Salpausselkä), and the upper part during the ice recession from the Ra.

During the accumulation of the brown clay drifting ice may have become very scarce, and was perhaps for a time quite absent as is suggested by a pebble-free zone, though this zone contains a small amount of displaced Foraminifera, which makes this point debatable. Possibly the beginning of the Boreal period is marked by the very marked increase in the Foraminiferal content which is apparent in the lower half of the brown clay (fig. 4). Accumulated ice-drift material, either coarse clastic deposits or displaced Foraminifera, is completely absent in the lower part of the foraminiferal marl, and this may mark the Atlantic period with its thermal optimum.

If it is assumed that the end of the Ra-period corresponds to a stratigraphic level which is between 40 and 50 cm below the surface of the core, it would mean a rate of sedimentation since the Ra-period of about 4–5 cm per thousand years.

A great deal more information is no doubt necessary before a more exact dating of the various stratigraphic units of the cores can be established, and also before an exact correlation between the different cores can take place.

ACKNOWLEDGEMENTS

The writer is happy to acknowledge the help and assistance given him during his stay at Scripps Institution of Oceanography. Special thanks are due to Dr. F. B. Phleger and Frances L. Parker at the Marine Foraminifera Laboratory. Thanks are also due to Dr. O. L. Bandy at the University of Southern California, for helpful suggestions with regard to the Foraminifera work.

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JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 29, NO. 1, PP. 30–37
FIGS. 1–6, MARCH, 1959

SMALL SCALE CROSS-LAMINATION IN LIMESTONES[1]

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ABSTRACT

Cross-stratification is a well-known structure in many detrital limestones. Several examples of very small scale cross-lamination in fine-grained limestones are described in the present paper, and these are cited as evidence of detrital origin. Cross-lamination in a limestone implies mechanical deposition of current transported particles and indicates that the limestone has not been precipitated in place.

Cross-lamination occurs on various scales in detrital limestones. In very fine-grained limestones, cross-laminae occur in "sets" of small dimensions, ranging in thickness from less than 1/25 of an inch to more than one inch, and in length from 1 to 2 inches to 6 or 8 inches. Small scale cross-laminae usually have angles of inclination from the horizontal of 20 degrees or less. Frequently, small-scale ripple marks occur with cross-lamination. Cross-stratification is also common in coarse, mechanically-deposited limestones such as calcarenites and limestone conglomerates. The maximum angle of inclination of cross-strata is greater in coarse grained limestones than in fine grained limestones. In calcarenites and in limestone conglomerates, the angles of inclination may range as high as 35 or 40 degrees from the horizontal.

Examples of small scale cross-laminations and of cross-bedding occurring in the upper portion of the Arbuckle Limestone of south-central Oklahoma are shown by negative peel prints and photographs in the present paper.

INTRODUCTION

It is not surprising that mechanically deposited limestones tend to show the same textures and structures as non-carbonate clastic sediments (Carozzi, 1952). Ripple marks, cross-bedding, cross-lamination, and graded bedding are displayed in many limestones. Lime muds and silts, as well as calcareous sands and gravels tend to behave much the same as their non-carbonate counterparts when deposited under similar conditions. Many geologists have noted the occurrence of cross-bedding in clastic limestones, as for example, in certain of the oölitic Pennsylvanian limestone units of eastern Kansas, such as the Bethany Falls Limestone member of the Swope Limestone (Moore, 1949). Much less attention, however, appears to have been paid to small scale cross-stratification, or cross-lamination, in limestones.

Observations on small-scale cross-lamination, particularly in fine-grained limestones, may be useful in determining the mode of origin of the limestones. Although the origin of the many fine-grained limestones is controversial, where such limestones display cross-lamination, there can be little doubt as to their clastic origin. It seems very unlikely that cross-lamination could be produced in a limestone which has been precipitated in place by chemical or biochemical means. Instead, a deposit created by mechanical transportation and settling of discrete particles is implied.

Excellent examples of cross-lamination may be seen in the Arbuckle Group of limestones of Cambro-Ordovician age, which crops out in the Arbuckle Mountains of south-central Oklahoma. Limestones of the Arbuckle Group include various types of mechanically deposited limestones, which include calcarenites, fine detrital laminated limestones, oölitic limestones, and limestone conglomerates. Non-detrital limestones include structureless pelitic limestones whose origin is poorly understood, and algal limestones (Ham, 1950, pp. 88–99). Some of the limestones contain various proportions of clay and silt; and dolomite is not uncommon, both as detrital grains and as replacements. Cross-lamination is locally common in the fine-grained limestones, and cross-bedding occurs in the limestone conglomerates.

Terminology

Terminology used in describing cross-lamination in ths paper is adapted from McKee and Weir (1953). They define cross-stratification as the arrangement within a

[1] Manuscript received June 16, 1958.

formation of layers at one or more angles to the dip of the formation. Thus, a cross-stratified unit within a formation is one with its layers deposited at an angle to the original dip of the formation. Cross-strata may be either cross-beds or cross-laminae, depending upon dimensions. A cross-bed is greater than 1 cm in thickness, and a cross-lamina is 1 cm or less in thickness. Cross-strata occur in "sets." A set may be defined as a group of essentially conformable cross-strata separated from other sedimentary units by surfaces of erosion, non-deposition, or abrupt change in character.

CROSS-LAMINATION IN UPPER PORTION OF ARBUCKLE LIMESTONE

In the figures accompanying this paper, examples of cross-lamination occurring in the West Spring Creek Formation of the Arbuckle Limestone Group are shown. The West Spring Creek Formation is the uppermost formation of the six formations into which the Arbuckle Group has been divided (Ham, 1950). All examples shown here were observed in outcrops along U. S. Highway 77, approximately four miles north of Springer, Oklahoma.

Figure 1 shows a complex group of sets of cross-laminae. The limestone is fine-grained and slightly silty. Differences in resistance to weathering, partially due to variations in silt content, cause the laminae to stand out in relief on the weathered edge of the limestone bed. The cross-lamination sets are of small size; they range from $\frac{1}{4}$ to $\frac{1}{2}$ inch in thickness, and extend not more than eight inches laterally. As appears to be typical in fine-grained detrital limestones, the angles of inclination of the cross-laminae are moderate and do not exceed 20 degrees.

Figure 2 is an example of the use of an acetate peel to show the details of very small-scale cross-lamination. This photograph is a negative print made by direct projection of the acetate peel in a photographic enlarger. This peel and others shown in succeeding figures were prepared by pressing polished surfaces of limestone specimens against an acetate sheet which had been wetted with acetone. Upon drying, the peels were carefully pulled away, providing an impression of the acid-etched surfaces and yielding a remarkable portrayal of petrographic details. In figure 2 the sets of cross-laminae are relatively thin, ranging from about 1/25 to $\frac{1}{8}$ of an inch in thickness; and they do not persist laterally for more than about one and a half inches. As is typical in the finest-grained limestones which show cross-lamination, the angles of inclination of the cross-laminae are relatively small, tending to be only 10 or 15 degrees from the horizontal, and frequently less, as shown in this example, where the cross-laminae are generally inclined about 5 to 10 degrees.

Figure 3 shows cross-laminae on a weathered vertical fracture surface. The cross-laminae occur in irregularly arranged sets which vary from $\frac{1}{4}$ to $\frac{3}{4}$ of an inch in thickness and are up to several inches in length. The sets create a series of crests and troughs which are rudely analogous to crests and troughs of ripple marks. In the upper part of the figure 4 the details of trough-filling sets of cross-laminae are shown. These cross-laminae are extremely thin, are composed of very fine detrital particles, and occur in sets which are about $\frac{3}{4}$ of an inch in thickness. The angle of inclination of the cross-laminae is about 10 degrees.

Figure 5 shows details of very small-scale ripple marks which are more regular in appearance than those previously shown. The ripple marks are shown in the lower third of the photograph. The ripple marks occur at the junction of fine-grained, relatively silt-free limestone (dark appearing, lower layer) and the overlying, slightly silty, fine grained limestone (light appearing). The silty limestone appears light because the photograph is a negative print in which the normal relations of light and dark are reversed. The ripple marks are slightly asymmetric and are about $\frac{3}{8}$ of an inch apart. The distance from crest to trough is slightly greater than $\frac{1}{16}$ of an inch. Cross-laminae which occupy the troughs have a low angle of inclination.

Cross-lamination is not confined to fine-grained limestones. Calcarenites are likewise cross-laminated, as shown in figure 6. As is typical in the more coarse-grained, detrital limestones, the maximum angle of inclination of the cross-laminae is greater than in fine-grained limestones. In the cross-

FIG. 1.—Typical appearance of small-scale cross-lamination of weathered vertical surface of a fine-grained slightly silty limestone. Cross-laminae occur in sets ranging from $\frac{1}{4}$ to $\frac{1}{2}$ of an inch in thickness and from 6 to 8 inches in lateral extent. Variations in attitude of cross-laminae suggest deposition by shifting currents. Ruler at left gives scale in inches.

FIG. 2.—Acetate peel showing very small-scale cross-lamination in fine grained limestone. Cross-laminae occur in sets ranging from about 1/25 to $\frac{1}{8}$ of an inch in thickness. Angle of inclination of cross-laminae generally between 5 and 10 degrees. Negative photographic print made by direct projection of acetate peel in photographic enlarger; magnification 3X.

FIG. 3.—Irregular cross-laminae appearing on weathered vertical surface of fine-grained limestone. Arrangement of cross-laminae "sets" creates a rude ripple-mark effect. Ruler at left gives scale in inches.

FIG. 4.—Acetate peel showing details of cross-laminae in fine-grained limestone. Cross-laminae sets in upper portion of photograph fill ripple mark troughs, are about $\frac{3}{4}$ of an inch in thickness, and have an inclination of about 10 degrees. Cross-laminae sets in lower part of photograph are thinner and have very low angles of inclination. Negative photographic print made by direct projection of acetate peel; magnification 2X.

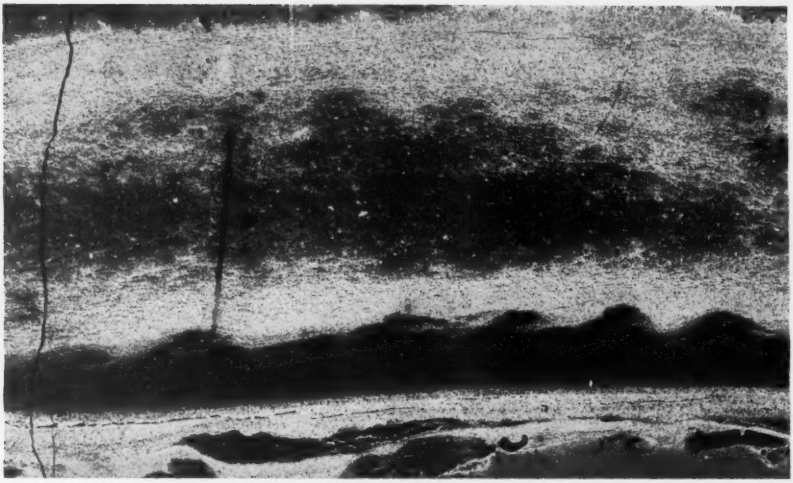

FIG. 5.—Acetate peel showing details of small-scale ripple marks in fine-grained limestone. Ripple marks occur at boundary between silt-free limestone (lower, dark appearing) and slightly silty, overlying (light appearing) limestone. Ripple marks are slightly asymmetric and are about $\frac{3}{8}$ of an inch apart. Distance from crest to trough is slightly greater than $\frac{1}{16}$ of an inch. Negative photographic print made by direct projection of acetate peel; magnification 2X.

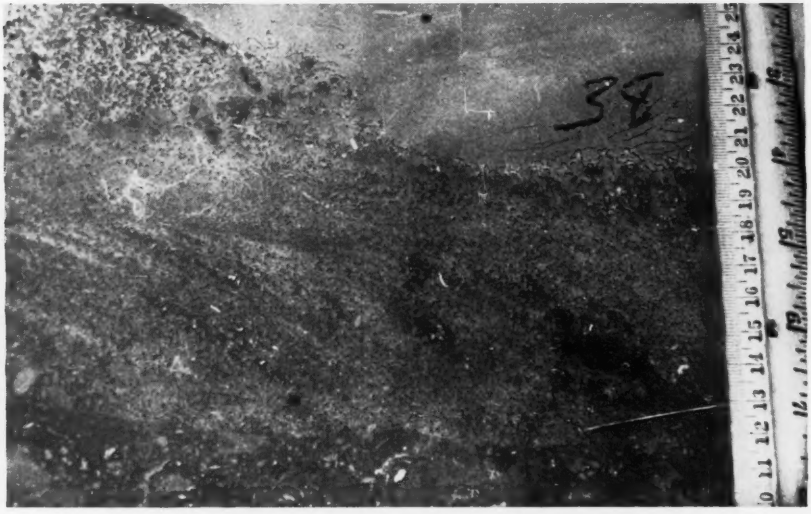

FIG. 6.—Set of cross-laminae in calcarenite on weathered vertical surface of bed. Angle of inclination of cross-laminae is about 30 degrees. Ruler at right gives scale in inches and in millimeters.

laminae shown in figure 6 the angle of inclination is about 30 degrees.

SUMMARY

In summary, it may be stated that cross-stratification is not uncommon in mechanically deposited detrital limestones. Since many limestones are of detrital origin, the presence of cross-stratification is probably widespread. Relatively large-scale cross-bedding in detrital limestones, as for example in oölitic limestones, has attracted the attention of many geologists. However, the presence of small-scale examples of cross-stratification, in the form of cross-lamination, is probably much more widespread than larger scale cross-bedding and should be looked for when detailed studies of limestones are made.

Frequent occurrence of cross-stratification in detrital limestones suggests that the limestones are deposited by currents in much the same manner as non-carbonate detrital sediments. It seems probable that many techniques of study previously applied to non-calcareous sandstones and siltstones can probably be applied to limestones. Perhaps the chief importance of cross-stratification in limestones is in fine-grained limestones where the presence of cross-lamination indicates that the limestone is a detrital deposit and not a chemically or biochemically-formed limestone which has been precipitated in place.

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JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 29, NO. 1, PP. 38–46
FIGS. 1–3, MARCH, 1959

SEDIMENTS FROM DANISH LAKES[1]

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ABSTRACT

The lake muds consist of three main components. 1. Organic matter (humus), 2. minerogenic matter, 3. an inorganic, biogenic component (diatom frustules and calcium carbonate).

For the investigation of such sediments chemical analysis is necessary, since common petrographic methods are not sufficient. The organic matter is best expressed as loss on ignition, the inorganic biogenic components as alkali-soluble SiO_2 and calcium carbonate, and the minerogenic component as the difference between total SiO_2 and alkali-soluble SiO_2.

A distinction between two types of humus is of greatest importance. This can be expressed as the content of organic carbon and by the carbon nitrogen ratio.

If the content of organic carbon is less than 50 percent and the C/N ratio less than 10 the humus is neutral, and if the content of organic carbon is higher than 50 percent and the ratio C/N is higher than 10, the humus is acid.

Triangular diagrams indicate the relations between lake types and sediments, and are also used to illustrate the development of two Danish lakes.

Organic sediments in lakes are like the uppermost A_0 horizon in soils, a mixture of minerogenic matter (sand and clay) chemical precipitates (particularly calcium carbonate) and more or less decomposed remains of dead plants and animals (humus).

Humus is a brown, grey or black substance whose chemical composition is still largely unknown, but it can be subdivided into two main types, acid humus and neutral humus (Rohhumus and Milder Humus respectively in German terminology).

From a colloid-chemical point of view, acid humus is presumed to be an unsaturated sol with negatively charged particles, and neutral humus (the humates) is thought to be a gel where anions are adsorbed to the surface of the humus particles. Soils with acid humus have been called Mor and soils with neutral humus have been called Muld (Müller 1887). The corresponding names of subaquatic soils (lake and bog deposits) are peat, dy and gyttja.

In peat, the destruction of dead plant material takes place in such a way that the original structure is preserved for a long time while at the same time unsaturated humus colloids (dopplerite) are produced.

In peat bogs, dopplerite forms lumps or networks in the peat. Fresh dopplerite is a black gelatinous substance with a specific gravity of 1.089. Exposed to the air it becomes first elastic and later hard and friable with a black-brownish color, a fatty-glassy lustre and chonchoidal fracture. The specific gravity is 1.466 and the hardness 2 to 2.5. It is insoluble in water, alcohol, and ether but totally soluble even in cold diluted alkali solution. It dries irreversibly.

Variations in chemical composition of dopplerite as determined by analysis of six samples are shown in table 1.

The terms gyttja and dy have been introduced by von Post (1862). Gyttja is a coprogenic deposit formed by animals on and within a lake bottom. It is a mixture of fragments of plants, diatom frustules, quartz and mica grains, siliceous spicules of *Spongilla* and several types of insect and crustacean exoskeletons together with pollen and cryptogam spores.

In a fresh state it is very soft and hydrous with a dark greenish-grey or black color, but never brown. In a dry state some gyttjas are hard and black, others are more friable and more nearly pale yellow-grey, depending on the main constituent.

Dy is a gyttja mixed with unsaturated humus colloids which are brought into the lake by streams draining nearby peat-bogs.

In a fresh state dy is also soft and hydrous but has a brown or brownish color. Dry dy is a very hard and slightly felty substance with a dark brownish to black color.

[1] Manuscript received July 11, 1958.

TABLE 1.—*Variations in chemical composition of dopplerite*

	I %	II %	III %	IV %	V %	VI %
C	55.46	56.46	52.00	57.65	48.06	57.09
H	5.48	5.48	5.00	4.62	4.98	
O	38.06	36.06	36.08	36.61	40.70	
N	trace		0.0	1.12	1.03	2.1
Ashes	5.1				5.85	3.5 –5

I. C. F. Nauman, 1885; II. Clarke, 1924; III. and IV. H. Potonie, 1911; V. and VI. Waksman, 1936.

TABLE 2.—*Classification of organic sediments by Potonie in 1908*

		Humulite (acid series)	Sapropelite (neutral series)	
	Terrestrial	Mor (Rohhumus)	Mull (Milder Humus)	
Recent		Peat Dopplerite	Dy Gyttja	Sapropel
	Limnic	Lignit (Browncoal)	Saprokol	
Fossil		Coal measures	Saprodil	
		Brigth coal	Sapantrakon Cannel coal Boghead coal	Oil
			Antracit	

From a pedological point of view, Potonie (1908) classified the organic sediments as shown in table 2.

This classification is based upon the nature of the humus and is entirely in agreement with the definition given by v. Post. The term sapropelite is perhaps not particularly good. Gyttja, even if it is deposited in lakes where the oxygen content in summertime is very low or even zero near the bottom, is quite different from the bluish-black, true sapropel with its content of H_2S and CH_4, deposited during strongly anaerobic conditions.

Dy also is called a sapropel-dopplerite by Potonie, which also is entirely in agreement with the definition given by v. Post. Its position in the system indicates that it is a gyttja mixed with dopplerite.

Later Nauman (1922) and Lundquist (1927) erroneously identified the gyttja with the organic substance in the sediment, and the dy with the unsaturated humus-colloids (the dopplerite). This erroneous definition unfortunately has been repeated in nearly all later publications (Iversen and Fægri, 1950).

The term tyrfopel was introduced by Nauman as a synonym for dy. It indicates a fine grained peat (torfpelit). However, this term can better be used for the bottom deposits in some smaller lakes where a rather large part of the original lake basin is covered by a quaking bog from which peat detritus has spread out over the whole lake bottom.

Standard petrographic methods (grain size analysis, heavy mineral analysis, etc.,) cannot be applied to investigations of these sediments. A microscopic investigation shows that they consist of three main components:

1. organic matter.
2. minerogenic matter (above all others, quartz grains).
3. an inorganic, biogenic component, mainly consisting of diatom frustules

TABLE 3.—*Alkali-soluble SiO_2 of some Danish clays*

	Total SiO_2 %	Alkali soluble SiO_2 %	Soluble SiO_2 in percent of total SiO_2 %
Eocene Lillebaelt clay	52.5	1.9	3.7
Miocene micaceous clay	57.1	1.2	2.1
Clay from ice dammed glacial lake	35.5	1.2	2.1
Clay from ice dammed glacial lake	44.1	2.1	4.8
Black clay from coal seams	59.2	2.3	3.9

and calcium cabonate originating from pulverized shells of mollusks and chemical precipitates from algae and the higher vegetation in the lake. (Wesenberg Lund, 1901).

The quantitative determination of these three components and of the humus type is possible only within certain limits of accuracy. In the writer's investigations the organic matter content has been taken to be equal to the loss of ignition. This certainly results in values that are somewhat too high, as some water loss from clay minerals is included. However, it is the writer's opinion, that loss of ignition is the best way to state the organic matter.

The organic carbon content can be determined more accurately by dry combustion. However the carbon content of the humus is essentially unknown. As shown later, the commonly used factors for conversion of carbon to organic matter are quite inapplicable.

Supposing that nearly all mineral grains are fine-grained quartz, this component could be determined as SiO_2. The total amount of SiO_2 however also includes diatom frustules. The latter however consist of opal, which is soluble in alkali. A determination of the alkali-soluble SiO_2 will, therefore, give the diatom content and the difference between total SiO_2 and soluble SiO_2 will approximately give the content of minerogenic matter. A small error is introduced by the limited solubility of clay minerals but as shown in table 3, which gives the alkali-soluble SiO_2 of some Danish clays, this error is very small. Even if the gyttja contains as much as 10 percent clay the error is of no importance.

The humus type can be characterized by the C/N ratio (carbon:nitrogen). In soils with neutral humus as tschernoshem and other arid soils and in certain groups of brown soils C/N is smaller than 10. (Anderson and Byers, 1934; McKibben and Gray, 1932; McLean, 1930; Leighton and Shorey, 1930). On the other hand peat and soils with acid humus have a lower content of proteins (Waksman, 1936) and Kivinen states (1934) that in different types of peat, C/N varies from 15 (Amblystegium and Cyperacean peat) to 35 (Sphagnum peat). In pure dopplerite C/N varies from 46 to 52.

In marine muds C/N varies from 8 to 12 (Debysser, 1955; Hansen, 1944; Waksman, 1933; van Andel and Postma, 1954) but van Andel and Postma state further that in the central part of the Gulf of Paria the C/N ratio is very nearly 5.6. Only in the deltaic part of the gulf where the mud contains partly destroyed remains of vegetation with a high content of cellulose and lignin does the C/N exceed 10.

Regarding lakes, Potonie (1937) mentions that in the sapropelitic mud of the Schollener See, C/N = 8.2 to 9.4.

From this and from investigations in Danish lakes it can be concluded that *if C/N is less than 10 the humus in the mud is neutral humus and the mud is a gyttja, and if C/N exceeds 10 the gyttja is mixed with acid humus and the mud is a dy or a tyrfopel.* Increasing values for C/N imply an increasing content of acid humus, ending with peat formation in a Sphagnum peat bog.

In limnology lakes are subdivided in two main groups (Thienemann, 1925):

I. Dy lakes with brown water and acid humus in the bottom deposits.
II. Gyttja lakes with colorless water and neutral humus in the bottom deposits.

The latter group is further subdivided in two subgroups:

TABLE 4.—*Analyses of lake muds from some Danish lakes*

		I %	C %	C % of I	N %	C/N	T %	M %	D %	CaO %	Fe_2O_3 %
Moerkesö	1	92.3	48.2	52.0	2.8	22.9	4.7	3.1	1.6		1.03
	2	88.7	46.2	52.0	2.1	18.7	3.8	1.9	1.9		0.76
Uglesö	1	81.8	40.9	50.0	2.5	16.1	12.2	9.4	2.8	1.5	0.9
	2	80.0	41.1	51.7	2.9	14.0	7.5	6.0	1.5	1.7	3.1
Hampen Sö	2	44.0	19	43.0	2.7	7.2	45.7	32.9	12.8	3.0	0.7
	3	46.0	21	45.5	2.2	9.8	47.9	32.7	14.2	2.0	1.7
	11	44.0	20.2	46.0	2.7	7.6	45.4	30.3	15.1	1.4	1.5
Slauensö	2	33.0	12.7	38.5	1.3	9.8	39.8	21.5	13.3	1.4	18.4
	3	23.0	11.4	49.5	1.4	8.1	40.9	25.5	15.4	2.0	36.9
Almind Sö	4	25.0	10.5	42.0	1.1	9.4	53.3	31.3	22.0	1.0	13.1
	8	23.8	9.8	41.0	1.1	8.7	53.2	20.7	32.5	0.9	12.3
	12	23.4	7.6	33.0	0.8	9.1	50.6	29.2	21.4	0.8	15.6
Borresö	2	28.0	10.9	39.0	1.3	8.5	44.6	25.2	19.4	2.3	16.0
	10	27.2	10.4	38.2	1.4	7.7	50.7	25.2	25.5	4.4	6.7
	17	32.9	10.2	31.0	1.1	9.0	48.3	25.8	22.5	4.7	6.5
Julsö	1	30.0	10.5	35.0	1.4	7.5	53.6	21.5	32.1	4.2	10.7
	5	30.3	10.9	36.0	1.5	7.2	52.6	15.6	37.0	4.9	6.8
	7	31.1	10.7	34.0	1.5	7.2	49.1	21.5	27.6	5.4	6.0
	8	28.5	13.4	47.0	1.6	8.4	47.2	26.2	21.6	2.7	6.7
Grane Langsö	2	14.7	4.3	29.8	0.6	7.8	70.6	69.0	1.6	1.1	3.5
	4	9.3	3.1	33.1	0.3	9.6	75.6	72.8	2.8	1.0	3.1
	5	11.1	2.4	31.2	0.1	8.8	76.2	73.8	2.4	0.9	2.7
Gribsö		32.0	19.6	61.2	1.3	15.1	51.4	42.5	8.9	$CaCO_3$ %	
Esrom Sö		34.6	10.2	30.7	2.3	4.5	31.4	28.5	12.9	24	
Slotssö		46.6	18.5	39.8	2.0	9.1	31.6	21.2	10.4	15.0	
Lyngby Sö	4	46.2	16.0	34.6	2.2	7.2	35.9	13.5	16.9	13.5	
	5	51.4	22.9	45.0	2.5	9.0	30.2	14.0	16.2	17.0	
	7	44.0	18.7	42.5	2.1	8.9	27.5	13.8	13.7	23.5	
Tystrup Sö	2		4.2				14.3	10.7	3.7	67.5	
	5		11.3				15.6	11.7	3.9	72.0	
	9		4.8				34.3	30.5	3.8	47	
Bavelse Sö	25		8.9				16.8	12.3	4.5	62	

I = loss of ignition. C = organic carbon. N = nitrogen. T = total SiO_2. M = minerogen matter (T ÷ D). D = alkali soluble SiO_2 (Diatoms).

a. Eutrophic (nutritive) lakes with a high production of plankton and with slightly transparent water.
b. Oligotrophic lakes (poor in nutritive matter) with only a sparse production of plankton and with very transparent water.

Table 4 gives analyses of lake muds from some Danish lakes. In three of the lakes C/N exceeds 10 and the bottom deposits in these lakes therefore contain acid humus. In the first two (Uglesö and Moerkesö) nearly half of the original lake basin is covered with a quaking bog now overgrown with fir, spruce, and bushes. From margins of the peat, partly decomposed plant remains and peat detritus are eroded and are distributed over the whole lake bottom. The sediment

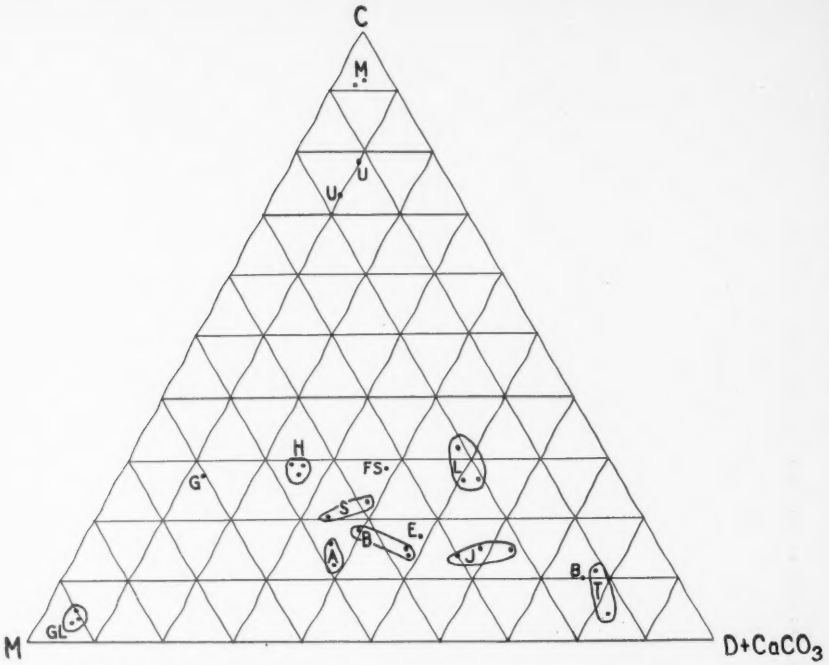

FIG. 1.—Triangular diagram showing the relationship between the three main components in the lake mud. In the uppermost corner the organic carbon (C), in the left corner the minerogene matter (M), and to the right the inorganic biogene component. ($D+CaCO_3$=diatom frustules and chemical precipitates.)

M=Moerkesö, U=Uglesö, G=Gribsö, H=Hampen Sö, FS=Frederiksborg Slotssö, L=Lyngby Sö, S=Slaunsö, A=Almind Sö, B=Borresö, E=Esrom Sö, J=Julsö, B=Bavelse Sö, T=Tystrup Sö, Gl=Grane Langsö.

is therefore a tyrfopel with acid humus and a high content of organic matter with a high content of organic carbon.

In Gribsö acid humus is transported to the lake by streams draining some peat bogs in the neighborhood (Berg and Petersen, 1956) and the sediment is a dy.

In all the other lakes C/N is less than 10. This indicates that the humus in the sediment is a neutral humus and the sediments are gyttjas. In these gyttjas the content of organic matter is rather low. In most of them it is lower than 30 percent. Only two have more than 40 percent ignition loss.

It must be noted that in all gyttjas the organic carbon content is less than 50 percent of the ignition loss. The same is also found in soils with neutral humus. This implies that the factors commonly used for conversion of carbon content to organic matter, 1.72 to 1.92 (Trask, 1939) and 1.887 (Waksman, 1933), are incorrect because they all assume that the organic carbon content of the organic matter is higher than 50 percent.

In the triangular diagram of figure 1, the lower right corner represents the eutrophic gyttja lakes with Tystrup Sö and Bavelse Sö as examples. The areas are 6.62 and 0.93 sq km respectively, and the greatest depth is 18 m in Tystrup Sö and 6 m in Bavelse Sö (Hansen, 1950). The river Susaa flows through the lakes and a strong decalcification of the water takes place in the lake (Berg, 1943).

In such lakes both production and de-

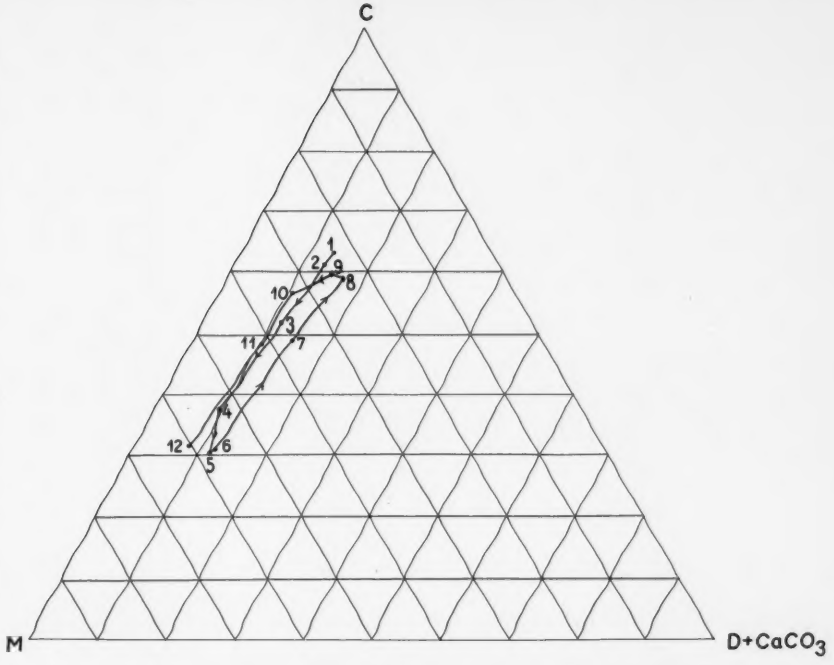

FIG. 2.—Triangular diagram showing the relationship between the three main components in the drilling in Grane Langsö.

composition or organic matter are high. In the bottom sediments the inorganic biogenic component dominates, especially the calcium carbonate originating from biogenic precipitation. The sediment is a lime gyttja or a lake marl (Hansen, 1950).

The left corner in the triangular diagram represents the oligotrophic gyttja lakes with Grane Langsö as an example. This lake is situated in the middle of Jutland close to the margin of the Würm (Wisconsin) glaciation, and it is surrounded by very sandy soil. The lake is 620 m long, 200 m wide and 11 m deep. The water is so transparent that a white disk can be seen at a depth of 11 m (Nygaard, 1955).

In such lakes the production of organic matter is very low and the minerogenic component dominates in the sediments. The sediment is a detritus gyttja.

Almind Sö, Borresö, Julsö, and Slauensö in Jutland and Esrom Sö in Seeland represent intermediate stages between oligotrophic and eutrophic gyttja lakes.

Hampen Sö is situated near Grane Langsö and is in many respects of the same type. The water is rather transparent and the gyttja is a detritus gyttja in which the minerogenic component is dominant, but the gyttja has a considerable organic matter content. The lake basin is a 1500 m long, 500 m wide and 13 m deep trough, with a 400 m long and 300 m wide, but only 2.5 m deep bay along the western side. In this bay, there is a very dense vegetation which produces an excess of organic matter as compared to the basin. This excess is great enough to disturb the balance between production and decomposition of organic matter. Consequently the gyttja becomes richer in organic matter than is usual in oligotrophic gyttja lakes.

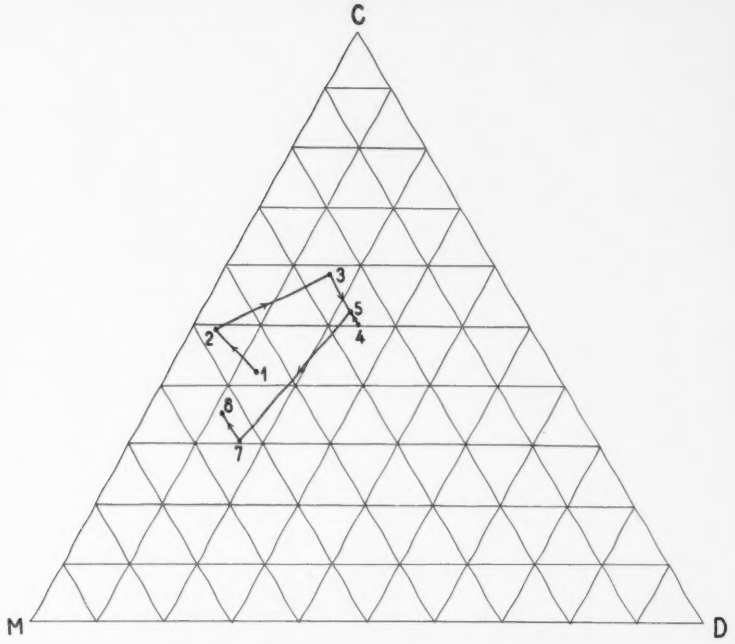

FIG. 3.—Triangular diagram showing the relationship between the three main components in the drilling in Gribsö.

The two Seeland lakes, Frederiksborg Slotssö and Lyngby Sö also have a high content of organic matter of the gyttja type. In these lakes a slight sewage pollution occurs. The lakes become super-eutrophic and the gyttja therefore is unusually rich in organic matter.

The upper corner in the triangular diagram represents the polyhumous lakes in which the sediments are rich in acid humus, which in turn is rich in organic carbon.

To facilitate study of the postglacial development of two of the lakes, drillings were made. In buried sediments the organic carbon content and the C/N ratio are not characteristic for the mud and the lake type, because very soon after the deposition diagenesis takes place which either goes in the direction of peat formation or of bitumen, resulting in rising values for the organic carbon content as well as for the C/N ratio because the proteins are the less resistant part of the organic matter (Twenhofel, 1950). Consequently, it is mainly the proportion of minerogenic and inorganic biogenic components which tells us something about the variation in lake type.

Figure 2 shows the results of borings in Grane Langsö. Every sample is 0.5 m long and small samples were taken 10 cm apart for pollen analysis.

In all samples the inorganic biogenic component occurs only in small but rather constant quantities (6 to 11 percent). In contrast to this the minerogenic component varies greatly. This means that the lake has always had the same oligotrophic character as today, but that considerable amounts of terrigenous material have been brought into the lake twice. The lowest samples (nos. 1 and 2) were taken from depths of 17.8 to 17.2 m and 17.2 to 16.7 m below the water surface (6.6 to 6.2 m and 6.2 to 5.7 m below the mud surface) and they were deposited

in Boreal time, Pollenzone V and VI (Jessen, 1935). These samples have a rather low content of minerogenic matter (12 to 13 percent). The same is the case for the samples 8 and 9 from 14.2 to 13.2 m below the water surface, the components of which were deposited in the Subboreal Period (Pollenzone VIII, Bronze age).

On the other hand, samples 5 and 6 from 15.7 to 14.7 m deposited in the Atlantic Period (Pollenzone VII), and sample 12 from 12.2 to 11.7 m from the Subatlantic Period (Pollenzone IX), have a high content of minerogenic matter (39 percent and 42.3 percent). Strikingly, these high values for the minerogenic component are found in both cases in wet periods. In the first case it is related to a rising of the water table in the lake. In the younger horizon the high content of minerogenic matter dates from a period from which pollen diagrams from peat bogs in other places of Jutland show layers of windblown sand.

The boring from lake Gribsö in Seeland (fig. 3) gives quite another picture of the development of a lake. Gribsö is 600 m long, 200 m wide and 10 m deep (Berg and Petersen, 1956). The deeper samples, no. 1 from 15.4 to 15.0 m and no. 2 from 15 to 14.5 m below the water table, were deposited in the Atlantic Period (Pollenzone VII). In this period the lake was nearly oligotrophic and the inorganic biogenic component of the mud was very low (2 to 6 percent). In samples 4 and 5 from 14 to 13 m, this component rises to 12 to 14 percent, implying that the lake in that part of the Subatlantic Period (Pollenzone IX) had a more eutrophic character. In the uppermost samples 8 and 9 the inorganic biogenic component again decreases to 9 to 10 percent and the lake is now a polyhumous lake with dy sediment.

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JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 29, NO. 1, PP. 47–55
FIGS. 1–5, MARCH, 1959

DENSITY DISTRIBUTION IN SEDIMENTS[1]

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ABSTRACT

The density of particles is a property which has not received adequate attention in recent studies of sediments. The distribution of densities in about 100 samples (chiefly sediments) was studied. Care was taken not to change the material by chemical means. The distributions are specific and reproducible for a given material. They confirm the evidence obtained microscopically that many sediments are made up of compound particles. In the samples studied very few particles with density greater than 3.4 or less than 1.4 were encountered. The method enables one to differentiate between younger and older sediments, as, during the aging of a sediment, organic matter combines (probably largely by physical forces) with mineral constituents, chiefly iron compounds. Below a particle size of 400 microns there seems to be no pronounced correlation between particle size and composition. The surface charges which bind the components into a composite aggregate may change on oxidation or on reduction, as shown by the change in density distribution. The method provides a useful extension to the existing approach to the study of sediments and shows that particle-size and particle-shape may not be the most important characteristics of a sediment.

METHODS

In the study of sediments attention is given chiefly to the *size* of disaggregated sedimentary particles under which circumstance the other important variable in Stokes' equation, the *density* of the particle, is assumed to be some convenient constant (as 2.65, the density of quartz). However, aggregated particles in sediments show densities ranging from 0.9–3.5 (excluding the minor free "heavy mineral" fraction), and a study of the aggregated grains seems desirable.

Microscopic examination of a sediment clearly shows the compound nature of particles. Iron compounds may cover quartz grains, or even penetrate into them. Organic matter may form coatings on mineral particles. Blue-green algae have been observed in various stage of pyritization. As the surface of the particles is the chief seat of microbiological activity, and as the influence of microbiological processes on sediments is great, there is added reason to study the nature of the aggregates.

The authors have examined the density-distribution of the constituent particles of sediments. These particles are left as much as possible in their natural state excepting that they are dried; no attempt is made to remove organic or acid-soluble matter. In highly reduced sediments the drying took place *in vacuo* to preclude oxidation.

In order to separate the samples into density-fractions use was made of methylene iodide, bromoform, carbon tetracholoride, benzene, and mixtures of two of these liquids. A complete range of densities from 0.88 to 3.36 can be made. Where hydrocarbons were suspected (as in coals) aqueous solutions of barium perchlorate giving a density range from 1.0 to 1.91 were substituted. Oven-dried or vacuum-dried material was separated into size-fractions by sieving. In most cases the fraction smaller than 100 microns was used. 100 mg aliquots were placed in small tubes of 5 mm internal diameter, containing liquids of known density. The amount that sank in one hour over a distance of 5 cm was separated, dried and weighed. No surface flocs were formed if the sample was sufficiently dry. In samples with a very high iron content there was a tendency for the particles to adhere to the wall of the tube. In such cases wider tubes (10 mm diameter) were used with success. Some very fine silts were centrifuged to effect better separation. In most cases, however, clean and rapid separation was obtained by sedimentation. When the weight of either

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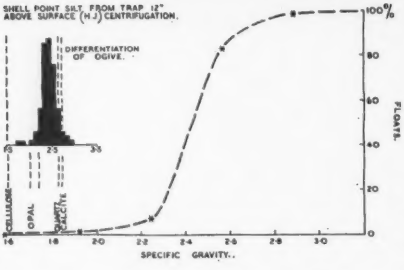

FIG. 1.—Density distribution shown by histogram and by cumulative curve.

the "sinkers" or "floaters" was plotted against density an ogive was obtained. On differentiation this yielded a histogram of the density distribution from which the mean specific gravity of the sample was determined (fig. 1).

The method is reproducible. Table 1 and figure 2 show the density distribution of two samples from an estuarine surface sediment at Shell Point, Georges River, New South Wales. The average density of the two samples is, therefore, 2.583 ± 0.009, and is accurate to two places in the second decimal.

MATERIALS

About one hundred density analyses were made of various materials obtained from the localities shown in table 2. These include terrigenous, alluvial fresh water, estuarine and marine sediments.

TABLE 1.—*Density distribution of two samples of estuarine sediment*

Density fraction	Sample A	Sample B
	%	%
2.9	0	1
2.8	16	17
2.7	26	26
2.6	26	26
2.5	12	12
2.4	8	8
2.3	4	5
2.2	4	3
2.1	2	1
2.0	2	1
Average density =	2.578	2.573

DISTRIBUTION OF DENSITIES

By pooling the frequencies of all the density-classes (n = 837) encountered in the 97 samples studied a composite curve is obtained (fig. 3). This curve approaches a normal distribution (shown in broken lines) but for a slight leptocurtosis (8% of the mean). In the same figure the density or range of densities of various sediment constituents is indicated. The values shown for cellulose, lignin, chitin and pectin have been determined by the authors, and those for several minerals were checked. The values extend from 1.2 to 3.5 with a mean at 2.407. Exclusion of coal and fossil limestone (the only pre-Quaternary sediments studied) narrows the range of densities, brings the distribution to near normal, and increases the mean density to 2.49 (see table 3).

As it is impossible to reproduce in this paper all 89 densitograms only a few typical examples will be given.

Types of Distribution

The distributions may be normal, bimodal, skewed (left or right), leptocurtic (excess at mode and mean), or bradycurtic (deficit at mode and mean), as shown in table 4. The figures for the bradycurtic distribution are too few to be of statistical significance. The mean density is alike for all other types, the bimodal distribution excepted. This is due to the high organic content of samples whose densities show bimodal distribution.

The amplitude of the distribution (the number of density classes) is lowest in pure

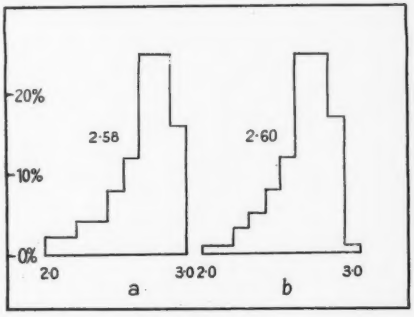

FIG. 2.—Density distribution of two samples of estuarine sediment.

TABLE 2.—*Densities of various sediments*

Locality	No. of samples	Material	Environment
Lake Macquarie, N.S.W.	14	Surface mud and core	Estuarine
Shell Point, Georges River, N.S.W.	17	mud	Estuarine
Gunnamatta Bay, Cronulla, N.S.W.	4	mud	Estuarine
Port Hacking River, Southwest Arm, N.S.W.	7	mud	Estuarine
Port Hacking River, Audley, N.S.W.	2	mud	Estuarine
Lake Conjola, N.S.W.	2	mud	Estuarine
Lake Coila, N.S.W.	2	mud	Estuarine
Lake Tuross, N.S.W.	2	mud	Estuarine
Lake Merrimbula, N.S.W.	2	mud	Estuarine
Lake Mallacoota, Victoria	6	mud	Estuarine
Pittwater, Tasmania	3	mud	Estuarine
Greenwood Bay, Western Australia	2	mud	Estuarine
Swan River, Cawley Bay, Western Australia	1	mud	Estuarine
Tasman Sea 100 miles east of Jervis Bay, N.S.W. (depth of 1000 m.)	2	mud	marine, terrigenous
Gouaro, New Caledonia	4	mud	coral reef
Oundjoh, New Caledonia	4	mud	coral reef
Isle Charron, New Caledonia	6	core	coral reef
Port la Guerre, New Caledonia	2	mud	freshwater
Lake George, N.S.W.	10	mud	freshwater
Wombat Moor, Tasmania	1	soil	freshwater
Canberra, A.C.T.	1	soil	alluvial
Coal Cliff, Wollongong, N.S.W.	1	coal	freshwater
Boulia, Queensland	2	limestone (Cambrian)	marine
Total	97		

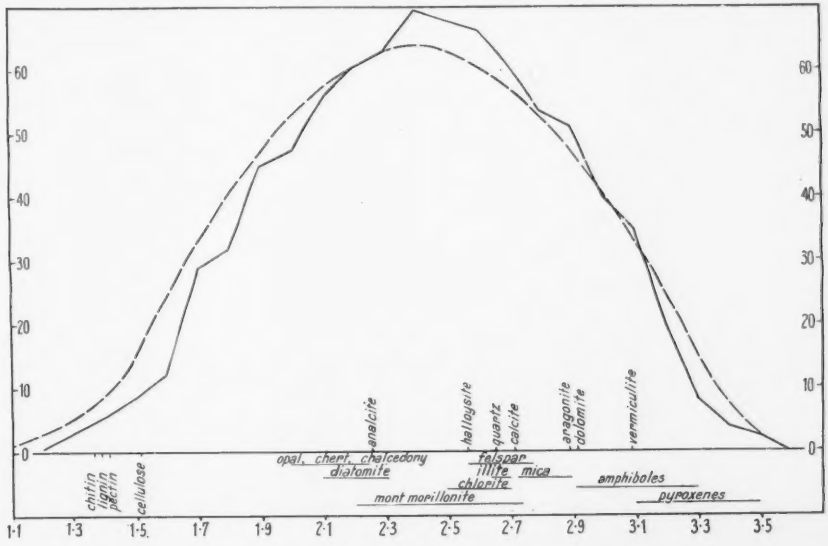

FIG. 3.—Composite curve showing densities of 97 samples listed in table 2.

TABLE 3.—*Distribution of 89 mean densities*

Density class	1.8	2.0	2.2	2.4	2.6	2.8	3.0	3.2
	0	10	21	31	23	4	0	
Normal distribution binomial quotient $(1+1)^8$	1.5	8	21	27	21	8	1.5	

or almost pure minerals, or in sediments consisting of particles of homogeneous composition. This accounts for the preponderance of the leptocurtic distribution. On the whole the amplitude seems to decrease with increase in average density.

From the determination of the iron content, expressed as FeO(OH), and of the organic matter the density of the remaining mineral fraction may be derived. From the data obtained, with additional determinations of the carbonates, we may classify the samples as in table 5. Certain distributions will be examined in more detail; see table 6 and figure 4. The bimodal curves are obtained usually from young muds. In slightly older samples a skew curve is obtained and this changes, in progressively older samples, to a leptocurtic distribution via a normal distribution. This sequence, which is not rigidly adhered to, may be correlated with gradual combination of organic matter with iron oxides and other minerals.

OXIDATION AND REDUCTION

The tendency of a particle to form aggregates must be caused chiefly by its surface charges. On the whole, particulate organic matter and quartz carry negative charges. Hydrated iron oxides seem to have a negative surface charge at high pH, but are highly positively charged at a low pH (Glasstone, 1951, p. 1215). Iron sulphides have a negative charge (Baas Becking, 1956). It stands to reason, therefore, that oxidoreductive changes in a sediment may lead to a disturbance of the structure. Both the total surface area and the amount of compound particles may change at every oxidation and at every reduction of the sediment. Such changes, seasonal and otherwise, take place in nearly every sediment (Baas Becking, Wood, and Kaplan, 1957). Densitograms are well adapted to describe such changes. Figure 5 shows the changes in density frequencies of eight sediments. Here the ordinates represent the change in density frequency (in percent) observed after reduction of an oxidized sediment. The reduced sediments were dried *in vacuo*, or the oxidized sediment was reduced by percolation with H_2S. In all cases there was a marked decrease in the number of lighter particles and an increase in the average density of the sample. A more systematic study of this phenomenon seems desirable.

TABLE 4.—*Types of density distribution as percentage of total*

Type of curve	Average density	Percent
Approaching normal	2.48	14
Bimodal distribution	2.35	22
Skew left	2.47	14
Skew right	2.47	10
Leptocurtic	2.48	37
Bradycurtic	2.73	3
Total		100

PARTICLE SIZE

In the natural sediment, determination of particle-size is of little significance, as most of the particles are compound. In about forty samples we studied the distribution of particle-size in the range 0–400 microns (clay to medium sand on Wentworth's

TABLE 5.—*Classification of samples*

Preponderant mineral	Percent of samples
Hydrated silica (opal, diatoms, sponge spicules)	30
Clay minerals and mixed	30
Quartz	24
Carbonates of calcium, magnesium, ferrous iron ("Lime")	16

TABLE 6.—*Classification of samples*

	Average density	Amplitude	Carbonate percent
a. *Calcite preponderant*			
1. Gouaro, New Caledonia, coral reef	2.70	10	54
2. Oundjoh, New Caledonia, coral reef	2.71	10	50
3. Gunnamatta Bay, New South Wales (300–400 microns)	2.75	7	58
Density of calcite	2.71		
			quartz %
b. *Quartz preponderant*			
4. Isle Charron, New Caledonia, surface sand	2.62	6	82
5. Port la Guerre, New Caledonia, surface mud	2.58	8	80
Density of quartz	2.65		
			clay %
c. *Clay minerals preponderant*			
6. Swan River, Crawley Bay, Western Australia, surface mud	2.48	7	64
Density of clay minerals	2.3–2.7		
			silica %
d. *Hydrated silica preponderant*			
7. Tasman Sea, 100 miles east of Jervis Bay, N.S.W. 1000 m. deep	2.12	6	80
8. Lake George, N.S.W., core from 3 ft.	2.29	4	94
9. Port Hacking River, Southwest Arm, N.S.W., surface mud	2.03	8	45
Density of hydrated silica	2.0–2.3		
			carbonate percent
e. *Lime plus organic matter and iron*			
10. Isle Charron, New Caledonia, core from 12 to 18 inches deep	2.77	8	30
			quartz %
f. *Quartz plus organic matter and iron*			
11. Pittwater, Tasmania, surface (200–300 micron fraction)	2.60	14	20
12. Gunnamatta Bay, N.S.W., surface (0–100 micron fraction)	2.63	20	20
13. Lake Mallacoota, Victoria, surface (0–100 micron fraction)	2.55	16	34
			Hydrated silica percent
g. *Hydrated silica plus organic matter plus iron*			
14. Georges River, N.S.W., surface, oxidized, fine fraction	2.31	13	not det.
15. Lake Conjola, N.S.W., surface, fine fraction	2.24	18	not det.
16. Lake Coila, N.S.W., surface, fine fraction	2.30	16	40
17. Georges River, N.S.W., surface, reduced, fine fraction	2.60	13	not det.

scale), without finding significant correlations between size and composition. Table 7 shows the densitograms of four size-classes in a surface mud from Gunnamatta Bay, New South Wales, where the finest fraction (only 4 percent of the total) contains all the organic matter. The coarser fractions, with a lime or quartz base, were devoid of organic matter. The fine silt deposited in a deep hole in the Port Hacking River showed an almost equal distribution of the organic matter in the four size-classes. Most of the heavy fraction was concentrated in the 100–200 micron class. The preponderant mineral here was hydrated silica in the form of diatom tests and sponge spicules.

ARTIFICIAL MUDS

Density analysis has been used by the authors to study the formation of compound particles in synthetic sediments. To a mineral matrix consisting of calcium carbonate, diatomite, quartz, kaolinite or mixtures of these substances, iron oxide was added. In one series the iron was added as cleaned steelwool, carbon tetrachloride being used for the cleaning. The organic matter added consisted of substances actually observed to

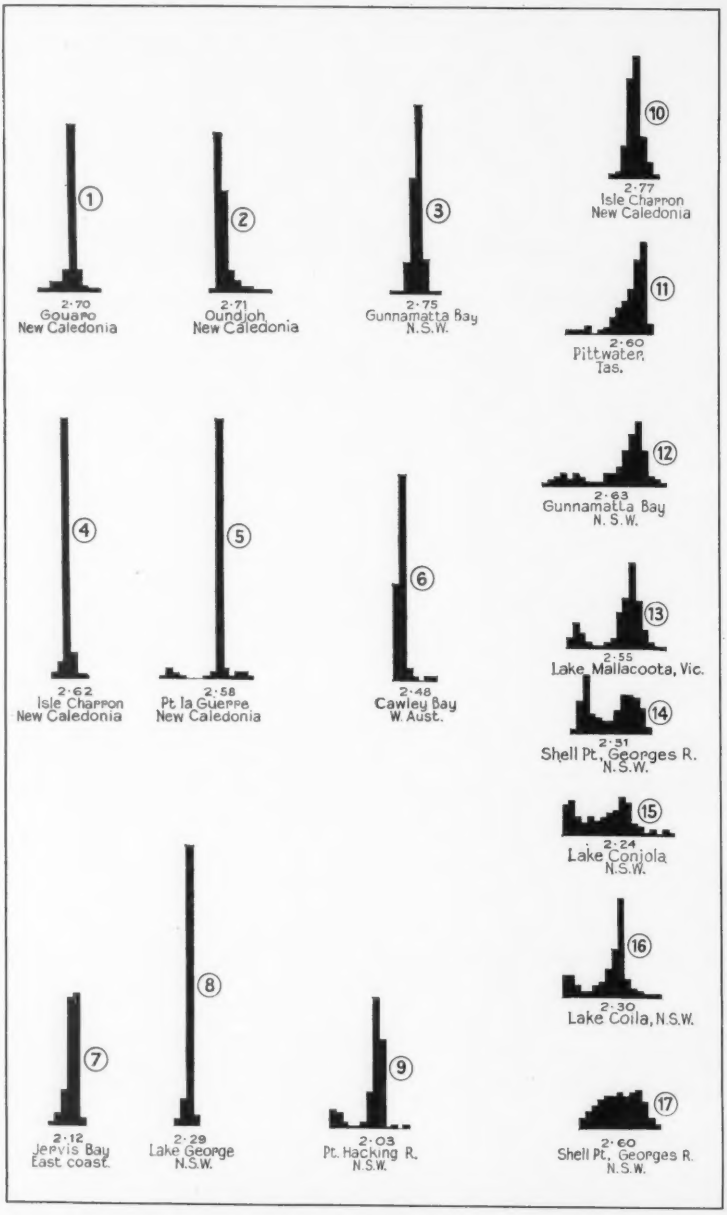

FIG. 4.—Histograms of density distributions of samples listed in table 6.

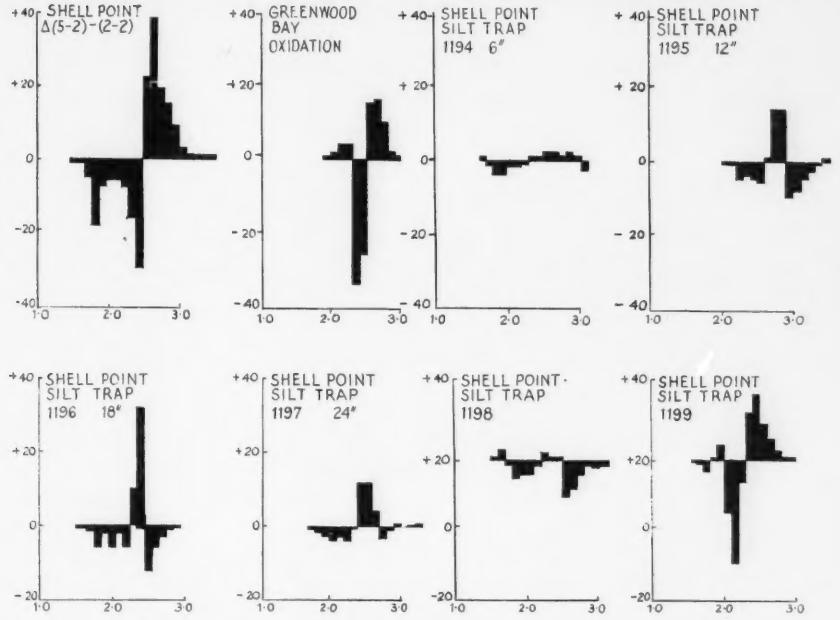

FIG. 5.—Changes in density frequencies of eight sediments shown in relation to oxidation-reduction potential.

occur in natural sediments in appreciable quantities. We used cellulose powder, hardwood lignin, chitin and pectin. The composition of the culture was, at the beginning: seawater, 1000 gms; black mud, 10 gm; solids, 100 gm, consisting of 6% haematite, 78% mineral matrix and 16% organic matter. Incubation was allowed to proceed in 250 ml screw-top jars in the dark at 20–25° C. The pectin cultures showed the characteristic black ferrous sulphides, products of sulphate-reduction, within a week, and the steelwool-cellulose cultures showed the same results after ten days. The other cellulose cultures remained negative for months. Both chitin and lignin proved unsuitable as media for sulphate reducers. Densitograms obtained five days after incubation still showed some sharp peaks at the expected densities. Table 9 shows the change in characteristics after 42 to 124 days incubation. The densitograms differ in no way from those observed in natural sediments. Though the composition of DKP remained constant between 110 and 124 days, the composition of LDP changed materially in this period. The work with artificial muds illustrated the consolidation of a sediment from its original components. Because the components are known (apart from the breakdown products of the organic matter) the study of synthetic muds should lead to a better understanding of the nature of the compound particles and of the oxidative changes in sediments.

DISCUSSION

The frequency distribution of densities in a sediment, the "densitogram," seems to characterize the material in more than one respect: age of sediments; amount of different particulate combinations, and approximate amounts of free and combined organic matter and iron. In only a few cases (iron hardpans) were particles heavier than methylene iodide (density 3.3) encountered.

TABLE 7.—*Density and particle size of two sediments*

	Gunnamatta Bay, Cronulla, N.S.W.				Port Hacking River, Southwest Arm, N.S.W., mud from 60-foot hole			
	lime muds				oxidized mud			
Density class	0 – 100 μ	100 – 200 μ	200 – 300 μ	300 – 400 μ	0 – 100 μ	100 – 200 μ	200 – 300 μ	300 – 400 μ
3.4								
3.3								
3.2	1	1						
3.1	2	2		1		1		
3.0	3	3		1		1		
2.9	11	7	14	11		2		
2.8	20	16	40	39		3		
2.7	16	31	34	36		3	2	1
2.6	11	30	10	10	1	1	3	1
2.5	6	2	2	1	0	1	2	0
2.4	4	1		1	1	1	3	0
2.3	4				0	3	4	2
2.2	1				28	23	18	26
2.1	1				42	32	35	36
2.0	1				11	14	15	17
1.9	3				2	7	5	5
1.8	4				1	3	3	1
1.7	2				1	2	4	1
1.6	4				2	2	3	2
1.5	3				5	1	2	4
1.4	2				6		1	3
1.3	1							1
Average density	2.63	2.64	2.75	2.75	2.03	2.15	2.10	2.04
% fraction	4	3	55	38	50	11	24	15

In all other cases the iron seems to have entered into combination with lighter material. The organo-iron combination will be described in another paper.

The classical approach to the study of sediments, concerned almost exclusively with the size, shape and mineralogy of particles, can be usefully extended by the study of density distributions, for size, shape and mineralogy of particles describe, in a way, only the skeleton of a living mud, even, after some of the cleaning techniques used, a "bleached" skeleton.

TABLE 8.—*Densities of materials used*

Symbol	Materials	Density
D	diatomite	2.1–2.3
K	kaolinite	2.61
L	lime	2.70–2.72
Q	quartz	2.65
St	steelwool	7.86
P	pectin	1.43
C	cellulose	1.50
—	haematite	4.9–5.3

The authors are indebted to Professor G. W. Leeper (Melbourne University) and to Messrs. H. Jitts and D. Izard (C.S.I.R.O.) for criticism and assistance. Mr. I. R. Kaplan collected and discribed the material from New Caledonia.

TABLE 9.—*Densities of artificial mud cultures*

Material	Incubation period (days)	Average density	Mode	Amplitude in density units	Density of lightest fraction
CSt	42	1.74	1.5	0.6	1.5
KP	42	2.59	2.6	1.2	1.9
DKP	110	2.41	2.5	1.1	1.9
DKP	124	2.39	2.5	1.1	1.9
LDP	110	2.50	2.5	1.9	1.6
LDP	124	2.62	2.6	1.4	2.0
LDP	124	2.75	2.7	1.0	2.3
PL	124	2.72	2.8	1.3	1.9
DP	124	2.38	2.4	1.6	1.6
QLP	124	2.74	2.7	1.2	2.0

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JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 29, NO. 1, PP. 56–63
FIG. 1, MARCH, 1959

CLAY MINERAL COMPOSITION OF SOME RECENT MARINE SEDIMENTS[1]

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ABSTRACT

The mineralogical studies on recent marine sediments collected in the western Pacific Ocean were carried out by means of microscopic, thermal, chemical, and X-ray methods. The 38 specimens which were studied can be grouped into muds, blue muds, red clays and globigerina oozes. Kinds of clay minerals were identified by X-ray, and their amounts were estimated quantitatively. Fragments of foraminifera, diatoms, sponges, quartz, and black opaque materials consisting of magnetite or a black manganese material are found in the sandy part. Illite is the principal clay mineral in all the specimens except globigerina oozes. Kaolinite and montmorillonite tend to be dominant in muds and blue muds (shallow-sea sediments). A chloritic mineral was also usually present in all the specimens except globigerina oozes, and is relatively abundant in red clays (deep-sea sediments). The chloritic mineral under consideration commonly decomposes at a temperature below 600° C, and as low as 450° C in some specimens.

INTRODUCTION

Recent advances in clay mineralogy have shed some light on the composition and properties of the finer-grained sediments. Among the investigations noted by the writers are the following: Grim, Dietz, and Bradley (1949), Aomine (1954), Suzuki (1954a and b), Kanematsu (1955, 1956), and Soveri (1956). Information concerning marine sediments is, however, still scanty. The present paper presents data on recent marine sediments collected from the western Pacific Ocean.

SPECIMENS

All of the specimens described here were supplied by the Hydrographic Office, Maritime Safety Board, Tokyo, Japan. They can be classified as red clays, globigernia oozes, blue muds, and muds.

MICROSCOPIC OBSERVATIONS

All the specimens are composed of clayey and sandy parts. Generally speaking coarse grains are more abundant in muds than in the other specimens. The sand grains can be identified as shown in table 1 after removing the clayey part by simple elutriation.

The writers particularly noticed black colored opaque grains, which are described in table 2 and are illustrated in figure 1.

[1] Manuscript received July 7, 1958.

ANALYSES

Differential Thermal Analysis Curves

Differential thermal analysis curves of the original specimens did not give clear thermal peaks except a very broad endothermic peak between 500° C and 600° C in red clays and muds due to clay materials, and remarkable endothermic peaks between 700° C and 800° C and/or between 850° C and 950° C due to carbonate minerals. For the purpose of confirmation of the presence of small amounts of montmorillonite and vermiculite minerals, differential thermal analysis was carried out on specimens pretreated by piperidine (Allaway, 1948). The presence of these minerals was suggested in some specimens by occurrence of very broad exothermic peaks between 600° C and 700° C.

Chemical Analyses

Seven selected specimens were analyzed chemically as shown in table 3.

X-ray Studies

X-ray powder reflections were examined by X-ray diffractometer. Strong peaks due to the minerals quartz, feldspar and calcite were confirmed. The former two are in general were abundant in muds and blue muds than in red clays. Calcite is particularly dominant in globigerina oozes. Generally speaking, a very broad band occurs which overlaps the peaks of the above minerals in

TABLE 1.—*Materials found as sandy grains in recent marine sediments*

Red clays	Large amounts of rounded yellow-colored shells of foraminifera, and black-colored, opaque, magnetic or non-magnetic grains. A small amount of fragments of diatoms
Globigerina ooze	Large amounts of shells of foraminifera. A small amount of diatoms and sponges. Large amounts of fragments of shells, and quartz grains, and black, opaque, magnetic fragments with irregular shapes

TABLE 2.—*Black colored opaque grains in recent marine sediments*

Shapes	Sizes (mm)	Magnetism	Occurrences	Observations on polished surfaces
irregular	0.1–0.5	magnetic	dominant in muds and red clays	magnetite
rounded	0.1–0.5	magnetic	in some specimens of red-clays	silicate minerals and magnetite crystals
sub-rounded	0.1–0.8	non-magnetic	in some specimens of red-clays	concentric texture; magnetite grains or foraminifera shells occupy its nucleus; its outer shell is composed of a black manganese material*

* It is very soft and can be scratched by needle and is easily dissolved in hydrochloric acid.

TABLE 3.—*Chemical analysis of recent marine sediments*

Specimen No.	17	1	T.10	9148058	9092018	9062003	9072015
	Mud	Blue mud	Blue mud	Blue mud	Globigerina ooze	Red clay	Red clay
SiO_2	53.72	59.45	57.84	48.66	25.86	54.42	54.59
Al_2O_3	18.90	14.91	17.08	11.74	7.07	14.73	11.49
Fe_2O_3	3.26	3.21	3.24	1.61	5.59	5.39	9.79
FeO	1.11	2.84	2.60	1.64	1.28	2.32	0.96
MnO	0.13	0.10	0.07	0.18	0.17	0.49	0.45
MgO	5.30	4.21	3.54	1.93	2.58	3.29	3.60
CaO	3.28	2.24	2.24	12.94	30.18	3.01	1.74
Na_2O	3.63	3.20	2.12	3.46	1.87	3.06	4.21
K_2O	1.80	2.30	1.49	2.30	0.52	1.33	2.72
TiO_2	0.60	0.60	0.54	0.55	0.35	0.70	0.62
H_2O	1.30	0.74	1.16	0.60	0.60	2.54	2.11
Ign. loss above 110° C	7.10	6.40	8.10	13.84	24.46	7.90	8.46
Total	100.13	100.20	100.02	99.45	100.53	99.18	100.74

the specimens except globigerina oozes. This banded reflection may possibly be due to amorphous materials.

Kinds of clay minerals can not be identified clearly, although the weak reflections of 15 Å, 10 Å and 7Å can be confirmed in some specimens. For the purpose of more detailed identification of clay materials, the following procedures were taken.

(*1*) *Well-oriented aggregate.*—The specimen was pulverized to a fine powder. A paste was made by mixing the powder with a few drops of water on a glass slide. By pressing this paste against the glass slide with a second glass slide, a thin film of the paste was spread over the glass surfaces. In this film of paste, the clay minerals were well oriented. Well-oriented aggregates usually

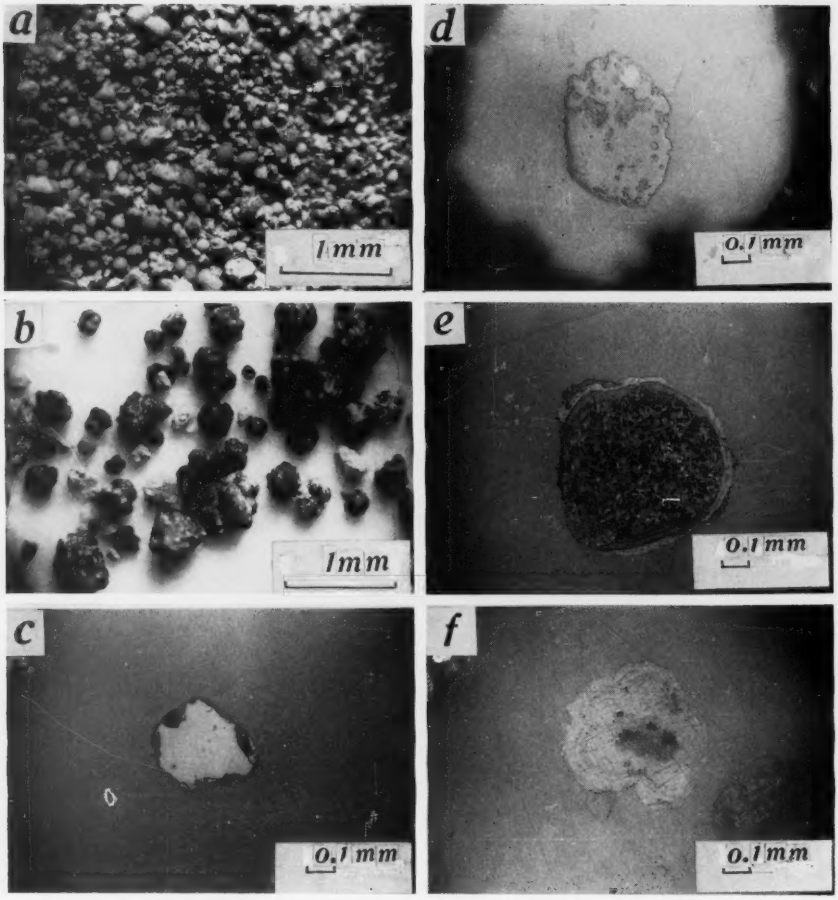

FIG. 1

a.—Coarse part of a red clay.
b.—Rounded black magnetic grains and sub-rounded non-magnetic grains in a red clay.
c.—Polished surface of a black, magnetic grain (magnetite).
d.—Polished surface of a rounded, black, magnetic grain (silicate minerals including magnetic crystals).
e, f.—Polished surfaces of sub-rounded, non-magnetic grains in a red clay. They have concentric structures: magnetite grains or foraminifera shells are found as nuclei and the outer shells are composed of a black manganese material.

are made from the clay fraction separated from an original sample by Stokes' method, but, in the present case, collecting a sufficient clay fraction is not always possible because of the small amount of each specimen available.

(*2*) *Treatment with ethylene glycol.*—The thin film over the glass-slide was saturated with a few drops of ethylene glycol and a thin film was reconstructed by pressing, the same procedure as mentioned above. As is well known, the presence of montmorillonite

TABLE 4.—*X-ray data for specimen No. 17 heated, or treated by ethylene glycol and hydrochloric acid*

Natural specimen		Ethylene glycol		450° C +H_2O*		450° C		600° C		HCl		
d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	
		17.3	8									M
15.2	8			14.7	8					15.8		M
10.0	8	10.0	5	10.0	9	10.0	13	10.0	8			I
7.25	20	7.25	13	7.69	10	7.25	23	7.31	10	7.25	10	K
				7.25	19							
				6.46	6			6.42	7			F
						5.01	5					I
				4.75	3	4.77	4					
4.484	6	4.504	10	4.504	10	4.506	11	4.484	13	4.506	12	I.M.K.
4.270	20	4.270	22	4.270	23	4.270	16	4.270	18	4.270	25	Q
4.060	18	4.077	22	4.058	15	4.070	17	4.058	20	4.040	20	F
		3.883	6	4.022	6	3.918	5	3.917	5	3.917	5	F
3.770	15			3.770	16	3.770	12	3.770	15	3.770	12	F
								3.663	10			
3.648	15	3.648	9	3.633	14	3.619	13			3.648	10	K
		3.548	5			3.548	5			3.520	5	I
		3.466	6			3.493	5	3.439	10	3.466	5	F?
3.351	70+	3.351	50+	3.351	120	3.351	65+	3.351	65	3.351	80+	Q
3.220	30	3.220	45	3.200	40	3.200	30	3.210	25	3.210	35	F

M: montmorillonite.
I: illite.
K: kaolinite.
F: feldspar.
Q: quartz.
* Specimen saturated with water after heating at 450° C.

was suggested when the 15.0 Å reflection is replaced by the 17 Å reflection, and the presence of hydrated halloysite is suggested when the 10 Å reflection is replaced by 11 Å reflection.

(*3*) *Heat treatments.*—The specimen was heated at certain temperatures for one hour, and the heated specimen was sealed in a glass tube immediately after cooling until it was radiated with X-ray.

Some earlier data show the noteworthy fact that the 14 Å minerals in marine sediments frequently behave as an intermediate mineral between chlorite and vermiculite or between vermiculite and montmorillonite (Johns, Grim, and Bradley, 1954; Bradley, 1955; Soveri, 1956). The intermediate mineral here means a clay mineral which behaves as mineral A in some properties, but as another mineral B in the other properties. It has been reported that a 14 Å chloritic mineral in marine sediments frequently decomposes at about 450° C or at a somewhat lower temperature than that of a well-crystallized chlorite which usually decomposes at about 700° C. Accordingly in the present case, each specimen after heating was examined at 450° C. and/or 600° C. In the case when heat treatment at 450° C caused diminishing of the 14 Å, 7 Å, and 3.5 Å peaks without replacement of the 14 Å peak by the 10 Å peak, the absence of kaolinite and montmorillonite, and also the presence of a 14 Å chloritic mineral is suggested. In a case where the 14 Å and 7Å reflections are persistent after heating at 450° C but diminish after heating at 600° C, the presence of kaolinite or a 14 Å chloritic mineral or both is suggested. The presence of kaolinite can be further confirmed after examining the change of ratio of 14 Å peaks to that of 7 Å peaks at elevated temperatures.

(*4*) *Treatment with hydrochloric acid.*—It has been anticipated that chlorite usually more easily decomposes by warming in dilute hydrochloric acid than do kaolinite and montmorillonite. In the present case, the reference specimens were warmed in hydrochloric acid (1:1) on water-bath. This procedure was helpful in discriminating kaolinite or montmorillonite from chlorite in the mixture of these minerals. Actual examples of procedures for identification of clay mineral compositions are as follows.

TABLE 5.—*X-ray data of specimen No. 9064546 heated, or treated by ethylene glycol and hydrochloric acid*

Natural specimen		Ethylene glycol		450° C +H_2O*		450° C		600° C		HCl		
d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	L	d (Å)	I	d (Å)	I	
		18.8	5									M
14.7	6	14.5	6	14.7	6	14.3	5					ch.M.
								12.3	5			
10.0	10	10.2	8	10.2	13	10.2	15	10.0	13	10.2	6	I
		8.51	7	7.69	8	8.6	8	8.5	5			
7.14	15	7.14	14	7.25	10	7.25	10	7.25	5	7.25	6	ch
6.42	6			6.42	6	6.51	6	6.46	6	6.51	6	F
						5.010	6	5.010	5	5.068	5	I
4.72	5											ch
4.506	8	4.506	5	4.506	7	4.506	8	4.506	10	4.506	10	I.M.
4.270	30	4.270	28	4.270	35	4.270	48	4.270	25	4.270	30	Q
4.040	20	4.040	15	4.058	15	4.040	18	4.058	13	4.058	20	F
3.883	8									3.883	5	F
3.770	13	3.770	10	3.786	15	3.785	19	3.786	15	3.786	15	F
3.677	10	3.677	6	3.678	10	3.678	12	3.693	9	3.663	12	
3.548	10	3.548	6	3.548	5							ch
3.466	7			3.493	7	3.493	10	3.493	8	3.480	10	I.F?
3.351	70+	3.351	70+	3.351	70+	3.351	65+	3.351	70+	3.351	75+	Q
3.209	35	3.200	60	3.209	50	3.209	55	3.209	55	3.209	45	F

M: montmorillonite.
I: illite.
ch: chlorite.
K: kaolinite.
F: feldspar.
Q: quartz.
* Specimen saturated with water after heating at 450° C.

Mud (*No. 17, table 4*).—Treatment with ethylene glycol: the 10 Å reflection is unchanged; the 15 Å reflection is replaced wholly by the 17 Å reflection. From this the presence of illite and montmorillonite and the absence of 14 Å chloritic mineral is suggested. A very strong 7Å reflection suggests the presence of kaolinite. Heat-treatment: the 15 Å reflection is replaced by the 10 Å, which is again replaced by the 15 Å reflection after the heated sample is wetted by water. From this, the presence of montmorillonite is suggested. Acid-treatment: the 15 A and the 7 Å reflections are substantially unchanged by this treatment.

Conclusively the clay mineral compositions of this specimen can be represented as illite, kaolinite, and montmorillonite. All the other powder reflections can be adequately explained also by the presence of these three kinds of clay minerals.

Mud (*No. 9064546, table 5*).—Treatment with ethylene glycol: The 14 Å reflection is replaced partly by the 17 Å reflection, but the 14 Å peak still remains unchanged as a small peak. This fact suggests the presence of a 14 Å chloritic mineral and also montmorillonite. The 14 Å reflection is persistent after heating at 450° C but is diminished after heating at 600° C, and also is diminished after acid treatment. This fact suggests the presence of a chloritic mineral, which decomposes at a lower temperature than 600° C. It can be expected that the 15 Å reflection due to montmorillorite can be confirmed after acid treatment, but actually it is not. Probably, in the present case, montmorillonite lattice may also decompose by the acid treatment. The presence of kaolinite can not be expected. Conclusively, the clay minerals in this specimen can be enumerated as montmorillonite, illite, and a chloritic mineral.

Red clay (*No. 9148036, table 6*).—The 14 Å and 10 Å reflections are substantially unchanged after treatment with ethylene glycol. This fact suggests the presence of a chloritic mineral and illite. The 14 Å reflection is still persistent after heating to 450° C. The 14 Å, 7 Å, and 3.5 Å reflections are diminished after acid treatment. Conclusively, the clay minerals in the specimen can be designated as illite and a chloritic mineral.

TABLE 6.—*X-ray data of specimen No. 9148036 heated, or treated by ethylene glycol and hydrochloric acid*

Natural specimen		Ethylene glycol		450° C +H_2O*		450° C		600° C		HCl		
d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	
14.7	8	14.7	7	14.3	8	14.3	5					ch
10.1	30	10.0	25	10.0	25	10.2	28	10.0	10	10.2	20	I
		8.4	8	8.5	6	8.5	10			8.5	5	
7.14	28	7.14	23	7.20	13	7.14	12	7.25	5			ch
						6.42	5	6.56	4			F
5.01	10	5.01	11	5.04	11	4.98	11	5.04	6	5.01	8	I
4.72	10	4.720	6	4.745	5							ch
4.484	8	4.484	6	4.506	6	4.484	8	4.506	13	4.484	10	I
4.270	20	4.270	17	4.270	20	4.291	20	4.270	20	4.270	30	Q
4.040	14	4.058	10	4.058	11	4.040	11	4.040	11	4.058	18	F
3.900	5	3.917	6	3.900	5	3.900	5	3.900	6	3.883	8	F
3.770	10	3.770	13	3.770	8	3.786	11	3.786	10	3.770	12	F
3.678	8	3.663	6	3.663	6	3.693	6	3.648	23	3.678	10	
3.548	20	3.548	16	3.534	11	3.534	8					ch
						3.493	8	3.507	6	3.507	8	I
3.351	65+	3.351	70+	3.351	65+	3.351	110	3.351	70+	3.363	70+	Q
3.209	35	3.209	40	3.209	35	3.209	40	3.209	28	3.209	25	F

M: montmorillonite. F: feldspar.
I: illite. Q: quartz.
ch: chlorite. * Specimen saturated with water after heating at 450° C.

QUANTITATIVE ESTIMATION OF CLAY MINERALS

The amounts of quartz, illite, montmorillonite, kaolinite and a chloritic mineral were estimated by X-ray. The method followed is that of Klug and Alexander (1954) using fluorite as internal standard. Standard curves were made by artificial mixtures of standard specimens and fluorite. It is very difficult to select the standard specimens agreeing with the clay minerals now under consideration with regard to their crystallinity; we can only carry here approximate estimations. The result is as shown in table 7.

CONCLUSIONS

Recent marine sediments collected from the western Pacific Ocean were investigated by means of microscopic, thermal, chemical and X-ray methods. The principal minerals were identified and their amounts were estimated quantitatively.

(1) Muds and blue muds: quartz and feldspar are more abundant than in the other specimens.

Illite is present, but its amount is less than in red clays. Kaolinite and montmorillonite are relatively more abundant than in the other samples.

(2) Red clay: The amounts of quartz and feldspar are variable. Illite and a chloritic mineral are the principal clay minerals, but montmorillonite is persent in some specimens.

(3) Globigerina ooze: The principal mineral is calcite. Quartz, feldspar and clay minerals are minor constituents.

As to clay minerals the following conclusions can be given. Kaolinite and montmorillonite tend to be abundant in muds and blue muds (shallow-sea sediments). Illite and a chloritic mineral usually were identified in all the reference specimens, but they are relatively abundant in red clays (deep-sea sediments). It is worthy of notice that the chloritic mineral decomposes at temperatures than 600° C commonly, and as low as 450° C in some specimens.

ACKNOWLEDGMENTS

The writers are greatly indebted to K. Suda, J. Yoshida, R. Komukai, T. Hoshimo of the Hydrographic Office for supplying valuable specimens and facilitating our studies. The writers also are grateful to H. Shibata, S. Okada, M. Tokunaga, M. Tatsu-

TABLE 7.—*Mineral compositions of recent marine sediments*

Sample number	Depth (m)	Quartz %	Feldspar	Calcite	Montmorillonite %	Chloritic material %	Illite %	Kaolinite %
17 mud	127	20	A	—	15	—	5	20
2 mud	250	20	A	—	10	?	5	20
9064546 mud	500	20	A	—	2–3	5	10	?
9090001 mud	23	10	B	—	—	5	10	—
9053320 blue mud	470	20	B	B	—	5	7–8	—
9148058 blue mud	1190	10	C	B	—	5	5	—
1 blue mud	150	15	A	—	10	?	10	15
9 blue mud	80	15	A	—	7–8	?	5	10
T 13 blue mud		20	B	—	10	2–3	7–8	—
T 31 blue mud		15	B	—	10	2–3	7–8	—
T 10 blue mud		15	B	—	10	2–3	7–8	—
T 15 blue mud		20	B	—	10	2–3	7–8	—
T 29 blue mud		20	B	—	10	2–3	7–8	—
88 gl. oz.	4494	2–3	C	A	—	—	—	—
9072022 gl. oz.	4365	2–3	C	A	—	—	—	—
9092018 gl. oz.	3012	2–3	B	A	—	—	—	—
A. 2751 gl. oz.	1256	2–3	C	A	—	—	—	—
9082006 gl. oz.	4196	10	C	B	—	—	—	—
9085041 red clay	4080	2–3	B	—	—	?	—	—
9148036 red clay	5498	10	B	—	—	10	30	—
9072015 red clay	5680	15	B	—	?	7–8	20	—
9023001 red clay	5704	15	A	—	—	7–8	20	—
9052054 red clay	4746	10	B	—	—	5	10	—
9148023 red clay	5243	7.8	B	—	?	7–8	15	—
9053312 red clay	4855	15	B	—	—	2–3	10	—
34 red clay	5508	15	B	—	—	2–3	15	—
9085050 red clay	5820	15	B	—	—	5	20	—
9072010 red clay	5737	10	B	—	—	2–3	10	—
9062003 red clay	6280	7–8	B	—	—	7–8	5	—
101 red clay	5913	7–8	B	—	10	2–3	2–3	—
9095006 red clay	4490	15	B	—	—	—	10	—
9072017 red clay	5760	10	B	—	—	—	7–8	—
9143015 red clay	4253	15	B	—	—	7–8	15	—
9072019 red clay	5345	5	B	—	—	5	7–8	—
9095004 red clay	5331	5	A	—	—	—	—	—
10 red clay	5015	5	C	—	10	2–3	2–3	?
9062030 red clay	5477	5	B	—	—	—	—	—
131 red clay	5881	7–8	A	—	—	—	5	—

A: much.
B: medium.
C: little.

moto, H. Hayashi, and H. Kodama of the Tokyo University of Education for valuable suggestions and criticisms of our experiments and considerations.

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JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 29, NO. 1, PP. 64–72
FIGS. 1–5, MARCH, 1959

PLAYA DEPOSIT IN THE BISHOP'S LODGE MEMBER OF THE TESUQUE FORMATION, SANTA FE COUNTY, NEW MEXICO[1]

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ABSTRACT

The examination of samples collected at two locations, one in the northern part of the Santa Fe Quadrangle and the other about 4 miles north, in the Tesuque Quadrangle, discloses the presence of amorphous gypsum in amounts ranging from 55 percent to 88 percent in the silts and sands of the upper beds of the Bishop's Lodge Member of the Tesuque Formation. Intense chemical weathering was noted in the volcanic and granitic components of the playa beds and the theory is offered that frequent wetting and dessication during deposition were responsible for mildly acidic solutions which could have caused the chemical weathering observed. The discovery of a marked disconformity at the top of the playa beds separating them from the overlying Tesuque Formation, coupled with changes in lithology, mode of sedimentation, and nature of terrain supplying the sediments, suggests that the Bishop's Lodge Member and beds underlying it should be separated from the Tesuque Formation.

INTRODUCTION

It has been suggested (Baldwin, 1956), that beds formerly included in the Santa Fe Formation should be known as the Tesuque Formation; the name "Santa Fe" being retained as a group name. The Bishop's Lodge Member of the Tesuque Formation comprises those beds which have been variously designated: "Picuris" and "Badito Tuff" by Cabot (1938), Abiquiu Tuff by Smith (1953), and "Abiquiu (?)" by Stearns (1953). In the Santa Fe seven and one half minute Quadrangle, the Bishop's Lodge Member is said (Baldwin, 1956) to be from 50–530 feet thick, and to rest comformably, in some places, on as much as 100 feet of Precambrian-derived sandstone typical of the Tesuque Formation. The composition of the Bishop's Lodge Member is described as light-gray volcanic-derived sandstone and silt, with minor tuff beds.

During the field season of 1956 the writer examined that part of the Tertiary section now designated the Bishop's Lodge Member of the Tesuque Formation, in the northeastern part of the Santa Fe Quadrangle and the adjoining Tesuque Quadrangle to the north (fig. 1). In the course of this examination a place was found where what apppeared to be an amygdaloidal basalt flow had been nearly destroyed by chemical weathering. All that remained of the flow were fragments of kaolinized basalt, the accessory minerals, and zeolitic material which had filled the amygdules of the flow. Evidence was sought in the enveloping beds for a possible cause of this phenomenon. Samples were collected, examined microscopically, and the mineral content determined; percentages were estimated by inspection.

When it was apparent that major percentages of the enclosing beds were in fact gypsiferous silts, the samples were submitted to Mr. Don Rawson, a Graduate Assistant in the Geology Department of the University of New Mexico for sieve analyses and microscopic study. From the data obtained it is apparent that the upper part of the Bishop's Lodge Member is a playa deposit. Neither the vertical nor lateral extent of the deposit can be defined at this time but the top of the deposit is exposed by the road-cut along the west side of the Bishop's Lodge-Santa Fe Highway (fig. 2).

PLAYA DEPOSIT

Two exposures of the playa deposit were examined in some detail, one in the Tesuque Quadrangle and one in the Santa Fe Quadrangle. They are separated by about 4 miles of rough terrain. In neither place was the bottom of the section exposed. The top of gypsiferous material, and the contact with

[1] Manuscript received June 9, 1958.

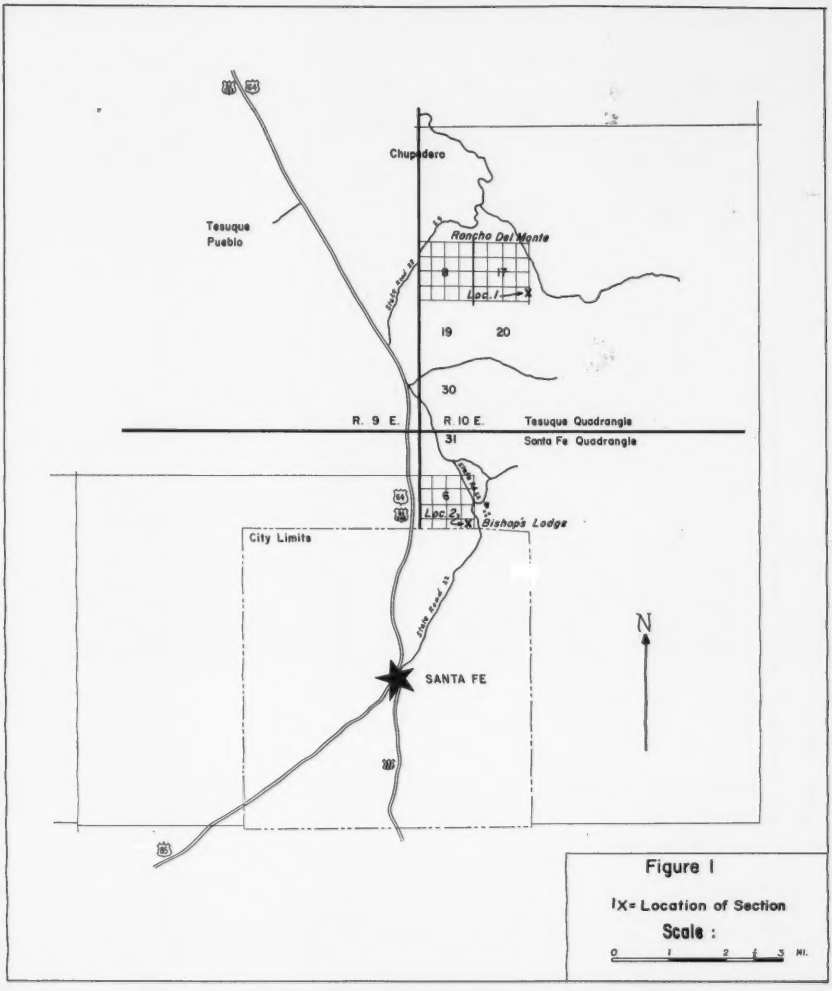

FIG. 1.—Map showing location of sections in relation to geography.

the overlying Tesuque Formation is well exposed at location 2 (fig. 1).

An interesting feature of the gypsiferous deposit is the relatively small amount of quartz present in the samples. For the most part quartz grains are subangular to subround, a few grains are iron stained and a few are frosted. Hornblende occurs usually as pseudohexagonal crystals and in considerable abundance, so much so that it imparts a slight "salt and pepper" effect to the outcrop of some beds. Feldspar, augite, muscovite, biotite, and magnetite occur in minor amounts, with the feldspars showing alteration to kaolin. The accessory mineral assemblage is such as would be expected from the disintegration of basaltic material. Shards of volcanic glass have not been

FIG. 2.—Santa Fe Quadrangle, South East Corner, Sec. 6, T. 17 N., R. 10 E. West side of Bishop's Lodge Road from Santa Fe. Contact between Tesuque Formation above and Bishop's Lodge gypsiferous silts below. Note difference between color and texture of beds above and below center of ruler. Boyer, '56.

recognized but may have been altered to clay minerals not recognizable by the methods of examination employed. Caliche in small amounts has been found in section 2, but does not seem to be present in section 1.

Basaltic grit, pebbles, and cobbles are intercalated with the silts in such a manner as to suggest deposition by currents of varying strength and volume. The coarser material is usually subangular to subround and for a deposit of this type the sorting is usually fair to good.

At Location 2, more than 100 feet of gypsiferous silts containing much basaltic material constitutes the upper part of the Bishop's Lodge Member. All the basaltic material, together with minor amounts of granitic material, have been subjected to intense chemical weathering. The result of this intense chemical weathering is easily observed in the outcrop and seems usable as a criterion for the determination of the position of the playa deposit where exposed.

STRATIGRAPHIC POSITION OF THE PLAYA

Although the total thickness of the playa beds cannot be stated for either of the locations examined, it is believed that at Location 2 it is more than 100 feet thick and constitutes the upper part of the Bishop's Lodge Member. It is separated from the overlying Tesuque Formation by a distinct disconformity. This disconformity may be responsible for the variations in thickness of the Bishop's Lodge Member as reported by Baldwin (1956), for areas to the south.

The Precambrian-derived, basal member of the Tesuque Formation as now defined,

SECTION NO. 1

Location: Tesuque Quadrangle; SE¼SE¼, Sec. 17, T. 18 N., R. 10 E. Near intersection of Hogback Trail from Rancho Del Monte, and wired gate in Forest Service fence.

Top of Section

		Thickness
1. Gray white gypsiferous silt		5′
Top of bed covered by granitic slope wash.		
2. Gray white gypsiferous silty basalt grit		3′
Outcrop slightly mottled, darker than beds above and below: many zeolites to 3 mm on surface.		
Rock fragments mostly weathered basalt	30%	
Amorphous gypsum	55%	
Quartz	10%	
Hornblende, magnetite, micas, accessories.		
Angular, sorting poor, predominant grain size 250–500 microns.		
3. Gray white gypsiferous sandy silt		7′
Gypsum, some may be anhydrite	85%	
Quartz, subangular to subround, crystals and grains, iron stained	8%	
Rock fragments and weathered basalt	2%	
Magnetite, muscovite, biotite, hornblende, very minor amounts of orthoclase. Well sorted, grain size ranges from less than 62 to 1000 microns; predominant grain size less than 62 microns.		
4. Gray white gypsiferous silt		5′
Gypsum	82%	
Quartz	5%	
Weathered basalt	5%	
Feldspar	2%	
Hornblende	3%	
Muscovite, biotite and magnetite minor.		
Well sorted, grains subangular to subround. Grain size range from less than 62–2000 microns; predominant grain size 62–125 microns.		
5. Gray white gypsiferous silt		20′
Gypsum	88%	
Quartz	6%	
Weathered basalt	2%	
Hornblende	1%	
Muscovite, biotite, magnetite and orthoclase in minor amounts. Subangular to subround; well sorted, grain size range from less than 62 to 500 microns. Predominant grain size 62–125 microns.		
Total		40′

The entire outcrop has a granular appearance in the field owing to the granular occurrence of the gypsum. The basalt fragments noted in 3, 4, and 5 are probably the remains of chemical weathering of basaltic grit or gravel. Neither top nor bottom of gypsiferous material exposed.

on which the Bishop's Lodge Member rests, is composed of gray-white to pinkish-white arkosic conglomerate, stringers of coarse, angular, granitic grits, sand, and intercalated beds of gray to pink silt. The pinkish cast of some of the beds is due to the amount of pink feldspar incorporated in the sediment. Gray granite, pink feldspathic granite, gneiss, occasional fragments of chert, quartzite, and gray limestone, cemented by calcarious silt, all apparently derived from the Sangre de Cristo terrain, are characteristic of this basal member. The angularity of much of the material, the poor sorting, and lenticularity of the poorly developed bedding suggest the mode of deposition as being intertonguing of alluvial fans. These apparently were deposited by rapidly flowing streams possibly aided by much slope wash from a low terrain to the east. Streams originating in the east have in a few instances left sorted, gravel-filled channels, with occasional shingled cobbles to attest to the principal direction of water movement.

The writer is inclined to view the lower member of the Tesuque Formation, as now defined, as a veneer of variable thickness laid down as a leveling process, filling the irregularities of the erosion surface developed on the Precambrian and Pennsylvanian rocks of the area. This filling

SECTION NO. 2

Location: Santa Fe Quadrangle; SE¼SE¼, Sec. 6, T. 17 N., R. 10 E., 700 feet southwest of Bishop Lamy's Chapel, on and under hill which splits abandoned dirt road from Santa Fe to Bishop's Lodge. (Fig. 3.)

Top of section—on west side of main highway out about 300 feet southwest of hill described above.

		Thickness
1. Gray white gypsiferous silty conglomerate		30′ ±
A gravel and pebble conglomerate composed of porphyry, basalt, granite and gneiss pebbles, irregularly bedded; lenses of pebbles surrounded by silts and finer grits. Pebbles excessively altered to Kaolin. Thickness uncertain, tie with bed 2 obscure, top of bed shown in Figure 2.		
Gypsum, mostly cleavage fragments	72%	
Rock fragments, weathered felsite porphyry and clay	4%	
Quartz, clear to frosted	6%	
Hornblende, pseudohexagonal crystals	4%	
Feldspar altering to kaolin	2%	
Magnetite and biotite; most of magnetite in 62–125 micron range 1–2%. Subangular to subround, sorting fair to poor, predominant grain size, 62–250 microns. Clay by decantation, 9%.		
Section on and under south side of hill with abandoned road, Figure 4.		
2. Gray white gypsiferous silty conglomerate		20′ ±
Boulders up to 18″ diameter, cobbles 2″–4″, pebbles and granules in streaks 6″–8″ thick separated by tannish streaks containing more clay. All small sizes and boulders are subround and are altered to kaolin. Porphyry, granite, basalt and gneiss probably original rock. Shape is retained but chemical weathering has destroyed component parts.		
Gypsum	65%	
Clay and ochre	15%	
Quartz	10%	
Caliche	4%	
Feldspar, partly or completely altered to kaolin	1–2%	
Subround, sorting is fair, grain size range, 62–500 microns, predominant grain size less than 125 microns.		
3. Gray white gypsiferous silt		8′
Not as coarse nor as well laminated as above.		
4. Gray white gypsiferous silt		
Gypsum	72%	
Clay and ochre	12%	
Caliche	4%	
Quartz	4%	
Magnetite	1–2%	
Biotite, feldspar, augite, muscovite, epidote—minor		
5. Gray white gypsiferous silt		17′
Section not well exposed, sluffing from road above obscures outcrop.		
Gypsum	77%	
Clay and ochre	12%	
Caliche	4%	
Quartz	3%	
Biotite, augite, magnetite, muscovite, hornblende—minor		
Mineral grains are angular fragments, some quartz grains are rounded and frosted. Weathered basalt and caliche occur in sizes above 500 microns. Sorting is good, predominant grain size is less than 62 microns.		
6. Gray white gypsiferous silty basalt grit		5′
Darker colored than beds 6 or 8. Many zeolites on surface. Rock fragments in sieving are angular pieces of basalt.		
Gypsum	74%	
Clay and ochre	15%	
Quartz	2%	
Caliche	5%	
Feldspar, mica, and magnetite—minor		
Grain sizes from 250 to less than 62 microns contain bulk of sievable material.		
7. Gray white gypsiferous silt		17′
Stringers of grit and small pebbles.		
Total		103′ ±

See note facing page ⇢

FIG. 3.—Santa Fe Quadrangle, Location 2, Looking North into valley of Little Tesuque Creek, along line between Sec. 6 and 7, T. 17 N., R. 10 E. Bishop's Lodge is about 700 feet North East of hill with dugway scar. Dugway is location of bed 2, Section 2, shown in Figure 4. July, '56.

material is definitely the product of the Sangre de Cristo terrain.

At the close of this leveling process, the area west of the Sangre de Cristo front must have presented the aspect of a relatively broad, gently undulating plain, across which the ancestral Rio Grande became the dominant sedimentational factor, bringing in material from an entirely different terrain. These river-borne sediments consitute the Bishop's Lodge Member.

The Bishop's Lodge sediments are radically different from anything else in the area. Basaltic and porphyritic material in abundance contrast strongly with the plutonic and metamorphic material on which it rests. Occasional stringers and small lenses of granitic grit and sand interbedded with the volcanic material suggest occasional dumping by torrential streams originating in the mountains to the east.

As sedimentation progressed in the developing basin west of the Sangre de Cristo front, a flood plain probably developed over a wide area north and south with eastward extensions reaching close to the Sangre de Cristo front as it exists today. This broad flood plain of the Rio Grande would seem to be an ideal environment for the development of shallow lakes or cutoff meanders

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Note: Below bed 7 there are about 140–160 feet of poorly exposed silts and clays with stringers of grit and small pebble conglomerate, to the unconformable contact with the Sandia Formation. Above bed 1, exposures are generally poor and covered with slope wash, but about 250 feet above is 15 to 18 feet of greenish black amygdaloidal basalt. There is also minor faulting in this 250 feet interval, upthrown to the west and the true interval cannot be measured at this point.

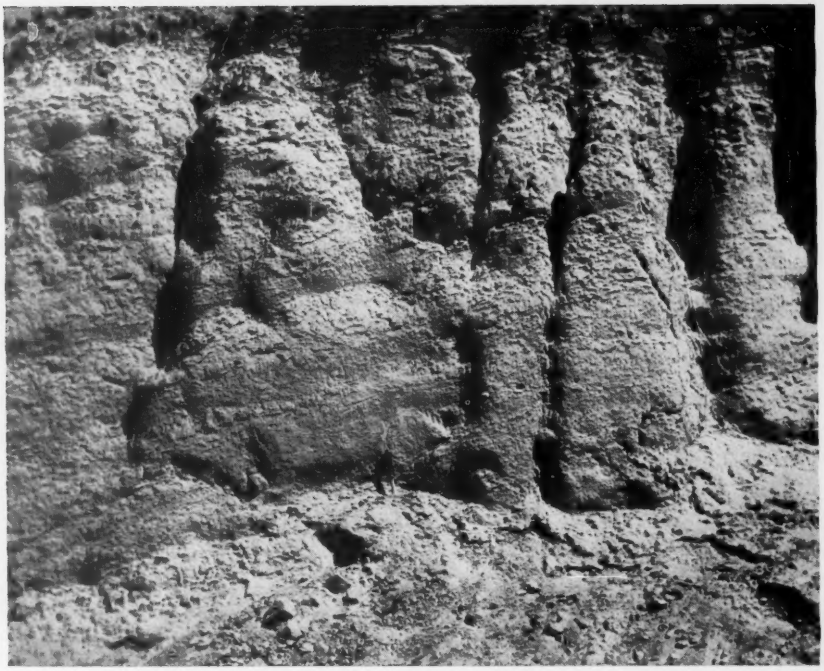

FIG. 4.—Detail of Bed 2, Section 2, Outline of porphyry boulder 10 inches in diameter above and to right of pick handle. Other boulders and cobbles of porphyry and basalt visible near by. Chemical alteration very intense. Drusy appearance of outcrop due to natural occurrence of gypsum plaster. Amorphous gypsum 65 percent. July '56.

which could be subjected to periodic flooding, sedimentation, and dessication. That flooding, sedimentation and dessication were frequent and that the waters were probably shallow, is attested by the amorphous character of the gypsum present in the playa beds. The development of lacustrine beds to the south and southwest reported by Stearns (1953), suggests that a relatively shallow lake may have developed behind some obstruction further south along the river, and may have extended into the Santa Fe-Tesuque area. Whether the playa beds found at locations 1 and 2 represent the flooding and dessication of shallow lakes, cut-off meanders, or the dessicated arms of a more widespread shallow lake is not certain; but the fact remains that such conditions did develop and playa deposits were formed.

Although the writer has not traced the playa beds between the two known exposures, field evidence suggests that the two exposures belong to the same sequence of beds.

The termination of the playa conditions at location 2 is marked by a definite disconformity shown in figures 2 and 5. The Tesuque Formation overlying the disconformity is composed of sediments transported from the Sangre de Cristo terrain and the intertonguing alluvial fans are again characteristic. The materials composing the fans do not seem to be as coarse grained as that observed in the basal Tesuque; they contain more clay and appear to have more intense color.

SUMMARY AND CONCLUSIONS

Two locations have been found where playa conditions can be demonstrated to have occurred during the late part of

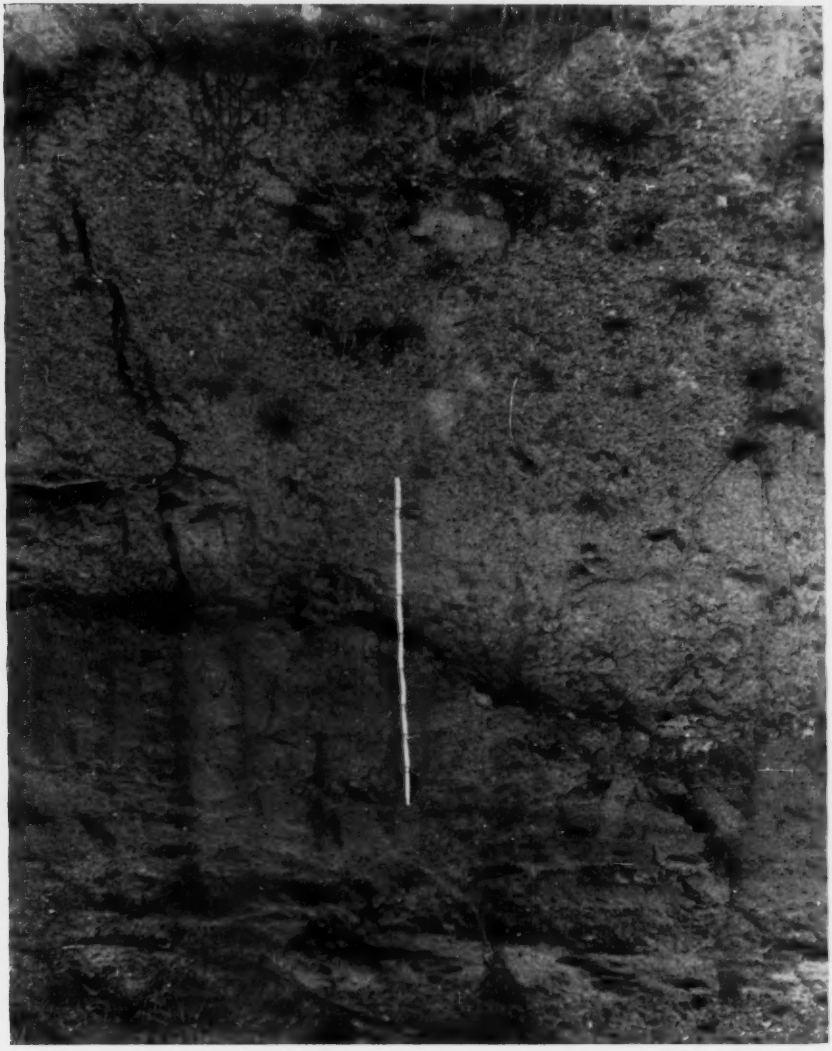

FIG. 5.—Detail of disconformity indicated in figure 2. Beds above center of ruler and above disconformity are light pink granitic grits, silts and conglomerate stringers typical of the Tesuque Formation. Below the disconformity are gray white silts and clays with volcanic debris representative of the Playa Member of the Bishop's Lodge. Amorphous gypsum and cleavage fragments 72 percent. July '56.

Bishop's Lodge time. Clays and silts are impregnated with amorphous gypsum to such an extent that the addition of water has probably produced a mild acidic environment which has attacked and altered the accessory minerals of volcanic and plutonic rocks entrained in the sediments. Although the extent, both laterally and vertically, of

the playa beds is not known, it is hoped that the recognition of playa conditions in the area will aid in the interpretation of the stratigraphic problems involved. The changes in lithology, sedimentation and terrain which supplied the sediments, within the basal part of the Tesuque Formation as now constituted, coupled with the strong disconformity at the top of the Bishop's Lodge Member, suggest that the Bishop's Lodge and the basal Tesuque of this area should be separated from the typical Santa Fe type beds comprising the Tesuque Formation.

ACKNOWLEDGMENTS

The writer wishes to acknowledge his indebtedness to Dr. Sherman A. Wengerd, University of New Mexico, for reading and criticism of the original manuscript and to Dr. Norman Hinds, University of California, for checking certain features of the field data.

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JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 29, NO. 1, PP. 73–76
FIGS. 1–3, MARCH, 1959

INSOLUBLE RESIDUE—MAGNESIUM CONTENT RELATIONSHIP OF CARBONATE ROCKS FROM THE DEVONIAN CEDAR VALLEY FORMATION[1]

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ABSTRACT

The insoluble residue content of carbonate rocks from several quarries in the Devonian Cedar Valley Formation of eastern Iowa is shown to be related to the mangesium content. The relationship of "soluble iron" content to magnesium content is plotted to demonstrate that a high soluble iron content is found only in rocks with a high magnesium content. The data presented may be of interest to persons concerned with the problem of dolomitization.

During an investigation of the chemical characteristics of some carbonate rocks as part of a research project aimed at determining the suitability of these rocks for concrete aggregate, an interesting relationship between the insoluble residue content and the magnesium content was noted (Bisque and Lemish, 1958). This relationship, based on quantitative measurements is worthy of consideration since it may be indicative of the factors involved in the genesis of the dolomitic rocks (Fairbridge, 1957).

Bulk samples representing lithologic variations were taken on a bed-by-bed basis from five quarries in the vicinity of Waterloo and Iowa City, Iowa (fig. 1). The quarries are situated in the Cedar Valley Formation of Devonian age. This formation (average thickness 80–100 feet) is subdivided into three members: the Solon, Rapid, and Coralville (fig. 1). The Solon Member is a massive, fine-grained, light-gray to buff limestone

[1] Manuscript received July 20, 1958.

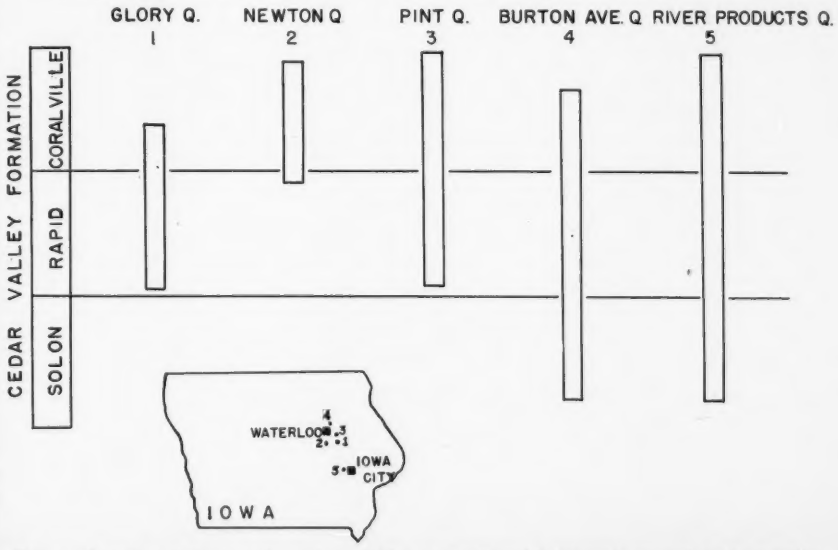

FIG. 1.—Location of the quarries and correlation of stratigraphic intervals exposed in the quarries.

with characteristic zones of black fossil fragments. This is overlain by the Rapid Member which consists mainly of a gray, argillaceous, well-bedded, calcitic dolomite with abundant chert nodules. The Coralville Member is generally a buff-colored rock and varies in composition both laterally and vertically from a calcitic dolomite to a lithographic limestone.

The insoluble residues were determined by dissolving a 2 gm sample of powdered rock in 3N hydrochloric acid for one half of an hour at 60° to 80° C, and separating the solids by filtration on a sintered glass filtering crucible. The residues were fine grained and found to consist essentially of chert, clay, and some pyrite (Lemish, and others, 1958). Clay minerals were separated from pulverized rock via a flotation technique designed to prevent damage to clay structures and then the minerals were identified by X-ray diffraction (Bisque and Lemish, 1958). The only clay mineral found was illite. The calcium, magnesium, and soluble iron contents were determined by titration with versene (ethylenediamine tetracetic acid) at appropriate pH levels as outlined by Cheng, and others (1952). The respec-

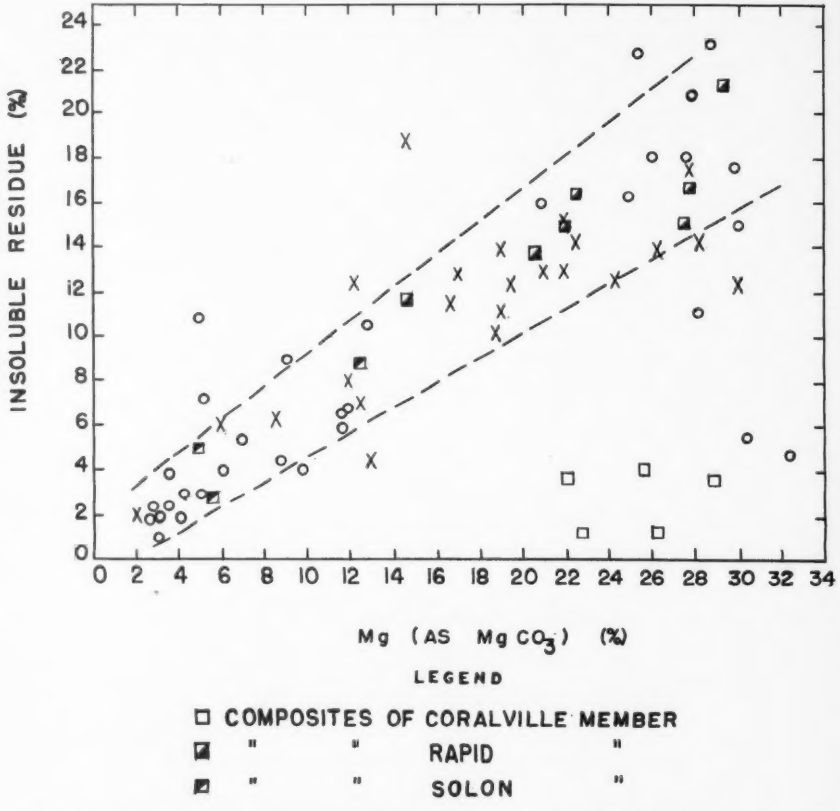

FIG. 2.—Analyses of composite, weighted samples of formation members from five quarries, including individual lithologic units of the Glory and Burton Avenue quarries.
X—Individual lithologic units of Glory Quarry.
O—Individual lithologic units of Burton Avenue Quarry.

tive calcium carbonate and magnesium carbonate content of each sample was calculated from these determinations. In figure 2, the magnesium content, as $MgCO_3$, is plotted against the insoluble residue content for samples from the five quarries. (See the explanation accompanying figure 2.)

The data demonstrate that the relationship of increasing Mg content to increasing impurities is well distributed through a fairly widespread range of rock types. A similar correlation was noted by Roy and others (1955), in their study of Mississippian rocks of Iowa. Dunbar and Rogers (1957, p. 223–224) in their recent text on stratigraphy discuss a similar relationship for Ordovician rocks from Pennsylvania as plotted by Lesley.

Petrographic thin sections were studied for each of the lithologic units investigated. The abundance of replacement dolomite rhombs correlated quite well with the amount of magnesium present as determined by the versene titration. In general the rocks with high magnesium content appear "dirty" in thin section due to the presence of many evenly disseminated opaque specks of non-carbonate materials. The data plotted in figure 3 illustrate the relationship of soluble iron content and magnesium content. It is interesting to note that a high soluble iron content never occurs except with an accompanying high magnesium content.

The values in the lower right-hand corner of figure 2 represent the Coralville Member which contains beds of porous, medium to coarse-grained dolomite in which nearly all primary features have been destroyed. These dolomitic zones have contacts which transgress bedding planes and which are interpreted as secondary features. The analyses of individual lithologic units of the Rapid Member and all the composite analyses as seen in figure 2 indicate that this member is characterized by a residue content greater than 10 percent and a $MgCO_3$ content 15 percent or higher. The validity

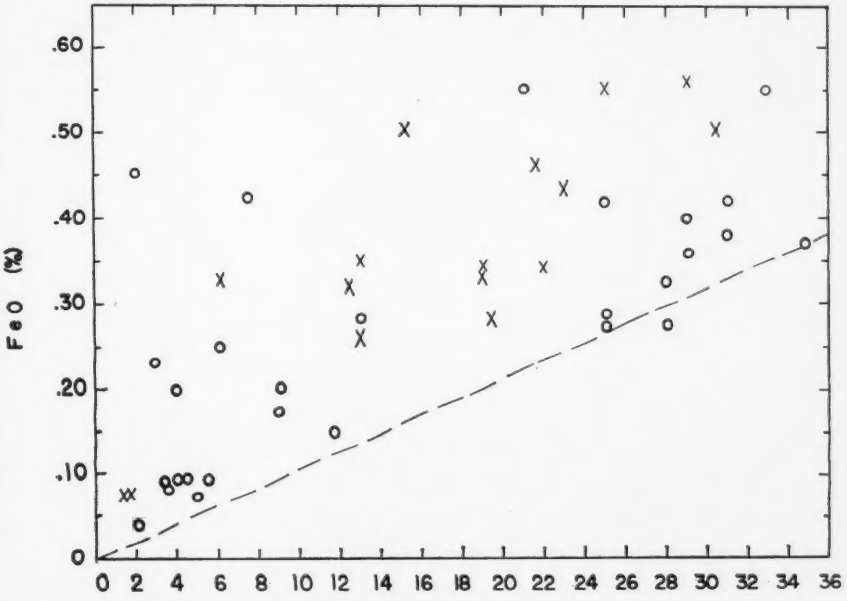

FIG. 3.—Relationship of "soluble iron" content to magnesium content.
X—Individual lithologic units of Glory Quarry.
O—Individual lithologic units of Burton Avenue Quarry.

of the separation of the Cedar Valley Formation into three distinct members on the basis of lithology is supported by the distribution of the data points representing analyses of composite samples of these members as seen in figure 2.

ACKNOWLEDGEMENTS

The data presented in this report represent a phase of the research conducted for the Iowa Highway Research Board with funds from the Iowa State Highway Commission.

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JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 29, NO. 1, PP. 77–86
FIGS. 1–6, MARCH, 1959

ANALYSIS OF INTERLAYER MIXTURES OF THREE CLAY MINERAL TYPES BY X-RAY DIFFRACTION[1]

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ABSTRACT

Much of the clay material in Recent and Tertiary sediments has been found to contain interlayer mixtures of three clay mineral types. An analytical method to determine the quantities of each clay mineral type in this kind of mixture involves gathering diffraction data from oriented aggregate specimens in equilibrium with 50% RH together with data from the specimen either after heating or solvating with ethylene glycol. The method provides precision commensurate with that of data which are obtained from this kind of clay material.

Recent and Tertiary sediments have been found to contain clay material which is difficult to describe in terms designed to be used for clays in older rocks. These young clays have been said to be *poorly crystallized* with respect to the clay in ancient sediments. The term *poorly crystallized* describes materials which give diffuse and weak x-ray diffraction patterns. Clays of this type can often be successfully analyzed in terms of interlayer mixtures of two or three clay mineral types that are characterized by different cell thicknesses. This paper describes a method for quantitatively determining from their x-ray diffraction effects the composition of interlayer mixtures in terms of three clay mineral components.

Interlayer mixtures of four clay mineral types undoubtedly occur. The quantity of the least abundant of the four would, however, be so small as to be justifiably ignored provided the quantity of each of the four does not approach 25 percent. It is felt that a method of interpreting diffraction data in terms of interlayer mixtures of three different periodicities is necessary but is adequate for studying poorly crystallized clays with four periodicities.

Procedures for identifying clay minerals from their x-ray diffraction effects have been well standardized for samples containing only one mineral. Values of $d_{(001)}$ are most useful for characterizing the four major clay groups—montmorillonite, illite, kaolinite, and chlorite. Higher orders of diffraction from the basal pinacoid are always integral submultiples of the $d_{(001)}$. Treatments resulting in uniform expansion or contraction of the mineral can be used as refinements of the identification technique. For example, heating montmorillonite causes its dehydration and contraction with a change in the basal d-spacing. The integral submultiple relation between $d_{(001)}$ and higher orders is preserved provided the entire sample undergoes a uniform change in unit cell thickness. Mechanical mixtures of two clay minerals produce diffraction patterns which are undistorted superimposed patterns of the component minerals.

The basic silicate layers of the three minerals illite, montmorillonite, and chlorite are structurally the same; their different interlayer compositions are used to distinguish them. An indirect indication of the interlayer composition of these minerals is found in the combined thickness of the basic silicate layer and interlayer material. The silicate layer (about 9.7 AU thick), plus one or two monomolecular layers of water, produces a basal periodicity of 12.4 or 15.4 AU, respectively, and is recognized as montmorillonite with Na^+ or Ca^{++} as exchangeable cations. The silicate layers which contain unexchangeable K^+ and no interlayer water are recognized as illite by their periodicity of about 10 AU. The silicate layers separated by interlayer $Mg(OH)_2$ produce a basal periodicity of 14 AU and are recognized as chlorite.

A second type of mixing, which has been called interstatification or interlayer mixing, is possible for illite, montmorillonite, and chlorite. It can be pictured as a single stack

[1] Manuscript received August 10, 1958.

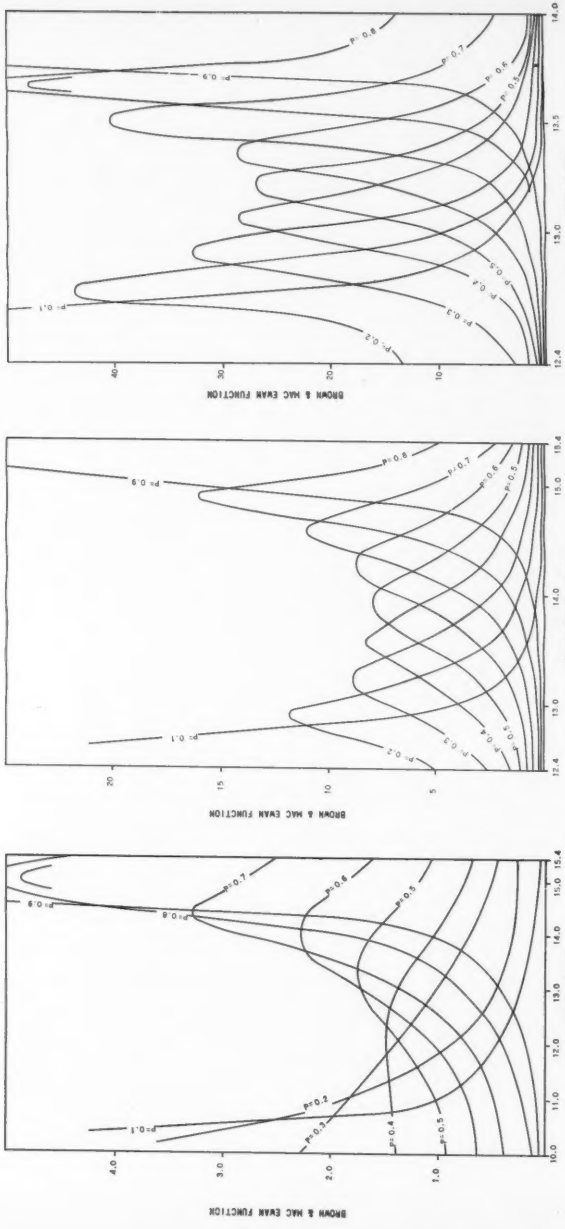

FIG. 1: a, b, c.—Curves of the Brown and MacEwan function plotted against apparent X-ray diffraction spacing, d', for interlayer mixtures of: a: 10 AU and 15.4 AU. b: 12.4 AU and 15.4 AU. c: 12.4 AU and 14 AU.

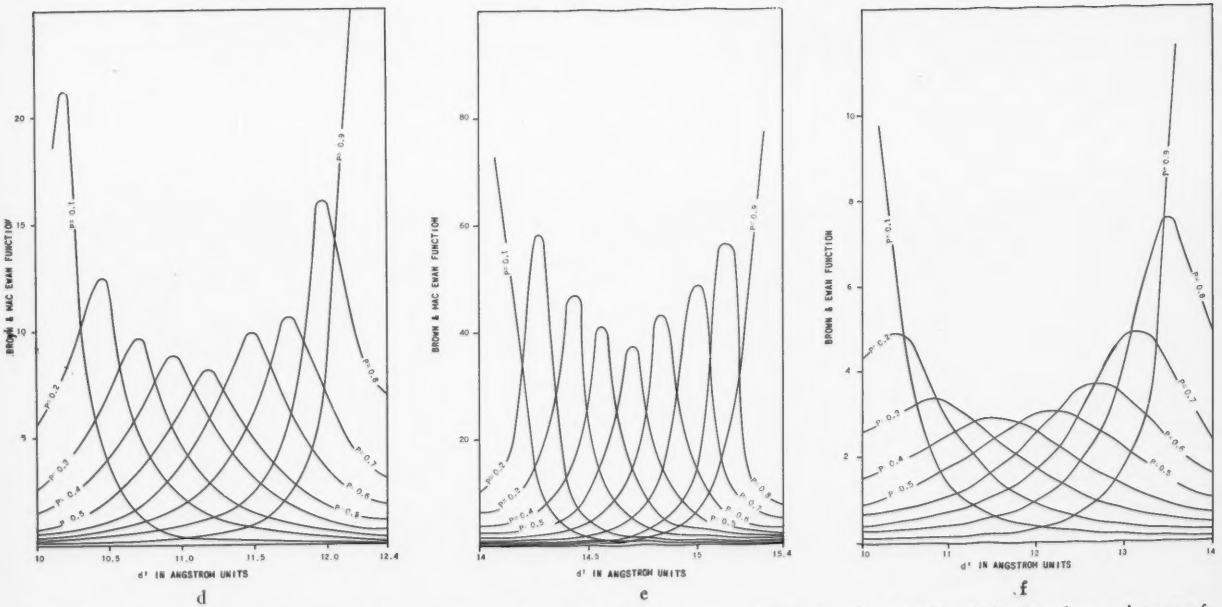

FIG. 1: d, e, f.—Curves of the Brown and MacEwan function plotted against apparent X-ray diffraction spacing, d', for interlayer mixtures of: d: 10 AU and 12.4 AU. e: 14 AU and 15.4 AU. f: 10 AU and 14 AU.

of the basic silicate layers separated by interlayer material of two or more kinds. Several consecutive silicate layers each of which is separated from the others by two monomolecular layers of water make up a zone which would be called montmorillonite. The succeeding silicate layers are not separated by water and would be recognized as illite. A repetition of this succession with the numbers of layers in each zone varying randomly would constitute an interlayer mixture of Ca-montmorillonite and illite.

The diffraction effects from an interlayer mixture are different from those produced by a simple admixture of the two clay types. Instead of two (00l) diffraction lines, one for each clay of the mixture, there is a single diffraction line located in an intermediate position. Adjacent lines of higher orders of (00l) will also coalesce. Hendricks and Teller (1942) have described the diffraction to be expected from a random mixture of periodicities. Brown and MacEwan (1949) have adapted the Hendricks and Teller (1942) general formula to the special analysis of random mixtures of only two periodicities. They have constructed theoretical diffraction patterns for random interlayer mixtures of 10 AU and 12.4 AU, 10 AU and 14 AU, 10 AU and 15.4 AU, 10 AU and 17.7 AU periodicities produced by (00*l*) diffraction from clays.

Diffraction intensities as calculated by Brown and MacEwan are the product of Ξ, F^2 (the structure factor), and the Brown and MacEwan function:

$$\frac{2p(1-p)\sin^2[\pi(d_2-d_1)/d']}{1-2p(1-p)\sin^2[\pi(d_2-d_1)/d] - p\cos 2\pi d_1 - (1-p)\cos 2\pi d_2/d'}$$

The values of Ξ can be obtained from

$$\frac{1-\cos' 2\theta}{\sin\theta \sin 2\theta} \quad \text{or} \quad \frac{1-\cos^2 2\theta}{\sin 2\theta}$$

depending on whether diffraction is being taken from a powdered sample or an oriented aggregate which simulates a single crystal. The experimental technique would obviously dictate which of these two functions would be favored. The problem of selecting values for F^2 is much more difficult because the structure factor varies with composition of the interlayer material and the silicate layer. The two types of basic layers involved in interlayer mixing probably have different structure factors. A compromise structure factor selected for any one combination of clays would probably not be as well adapted to a second sample with a new combination of clays.

The Brown and MacEwan function is specific in terms of p, the proportion of layers with the higher periodicity, and d', the apparent spacing of the random interlayer mixture. Curves of the Brown and MacEwan function for first-order diffraction from mixtures of 10 AU and 12.4 AU, 10 AU and 14 AU, 10 AU and 15.4 AU, 12.4 AU and 15.4 AU, 12.4 AU and 14 AU, 14 AU and 15 AU are shown in figure 1, a-f. These curves are included to enable each investigator to make his own selection of F^2 and Ξ and with little effort draw intensity curves for his specific sample and technique. Brown and MacEwan (1951) went on to multiply this function by their selection of Ξ

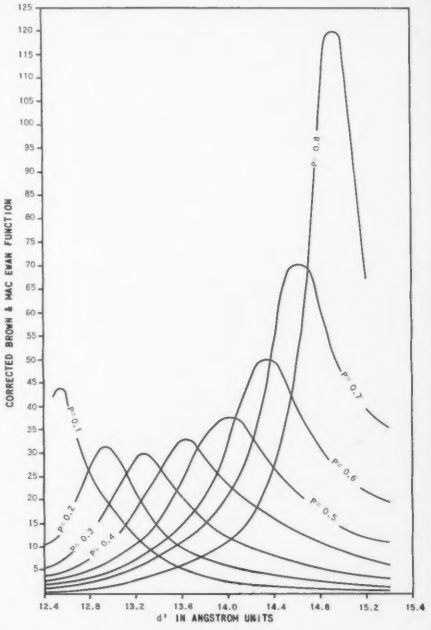

FIG. 2 Curves of the corrected Brown and MacEwan function plotted against apparent X-ray diffraction spacing, d', for an interlayer mixture of 12.4 AU and 15.4 AU.

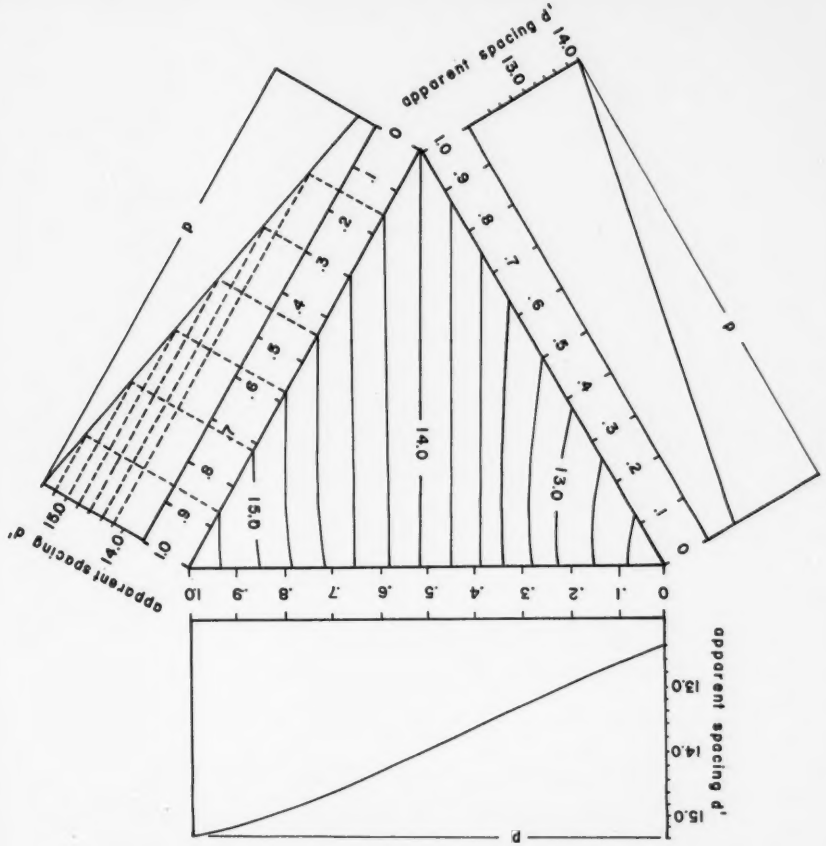

FIG. 3.—Three-component composition triangle for 12.4 AU, 14 AU, and 15.4 AU with contoured apparent X-ray diffraction spacing, d'.

and structure factor to obtain the set of general working curves which they published. Unless something specific is known about the composition of the clay which is forming the interlayer mixture, the general curves published by Brown and MacEwan (1951) are adequate. There are three possible combinations of periodicities that were not considered by Brown and MacEwan, namely, 12.4 AU and 15 AU, 12.4 AU and 14 AU, 14 AU and 15.4 AU, one of which is shown in figure 2. This curve is of intensity values for (00l) plotted against d', the apparent periodicity of the interlayer mixture. Intensities were obtained by multiplying the Brown and MacEwan function by compromise values of F^2 and Ξ similar to those selected by Brown and MacEwan for their combinations of periodicities.

The calculated diffraction maxima for (00l) for various proportions of Na-montmorillonite and Ca-montmorillonite with 12.4 AU and 15.4 AU periodicities are shown in figure 2. As Brown and MacEwan have shown for all of their combinations of periodicities, as the quantity of 15.4 AU

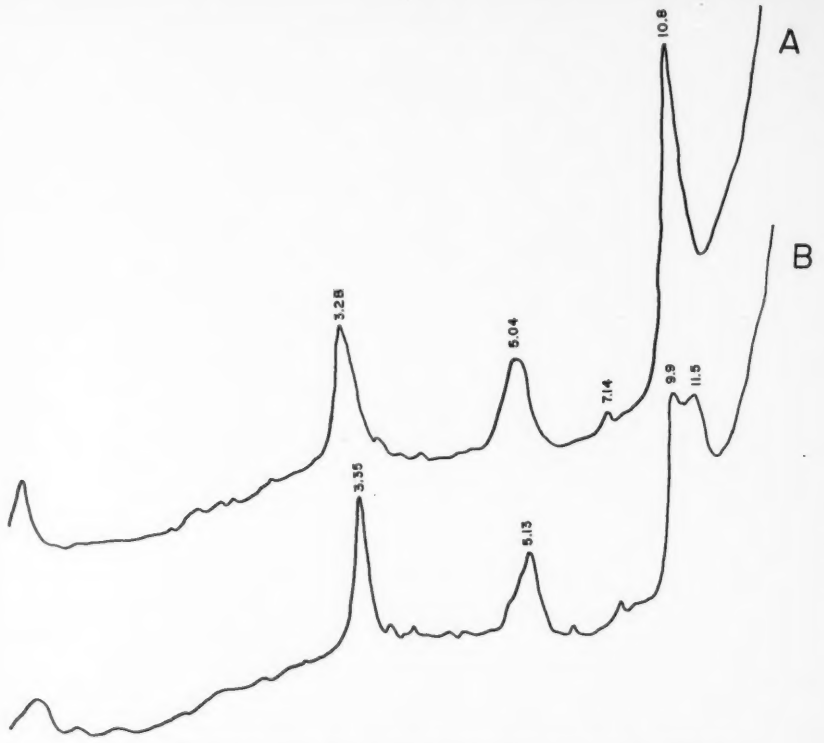

FIG. 4.—Diffraction patterns from oriented aggregates of a clay in equilibrium with 50% RH (a) and treated with ethylene glycol (b).

material increases, the d' at maximum intensity changes progressively away from 12.4 AU. This shifting of d' is not exactly proportional to the quantity of 15.4 AU layers in the interlayer mixture. The variation of d' at maximum intensity of mixtures of 12.4 AU layers with different proportions of 15.4 AU layer is shown in figure 3. Similar curves for the combinations of 12.4 AU and 14 AU and 14 AU and 15.4 AU minerals are also shown on the other two legs of the triangle (fig. 3).

Another prominent feature of the curves in figure 2 is the reduction in diffraction intensity for mixtures of nearly equal proportions of two periodicities. Diffraction from randomly interstratified mixtures of two mineral types is characterized by weakness and diffuseness. Brown and MacEwan point out that $d_{(001)}$ values for higher orders are not integral submultiples of the $d_{(001)}$. These three features, diffuseness, weakness, and irregular values of $d_{(001)}$, either collectively or individually are suggestive of interlayer mixing.

At higher angles where coalescence of more diffraction maxima is likely, diffuseness and low intensity are even more detrimental to analysis than in the (001) region. The analytical procedure described herein is concerned only with (001) diffraction. In the experience of the authors, the material to which the methods are applicable has such poorly developed higher orders as to be useless.

Figure 4 is a pair of diffraction curves

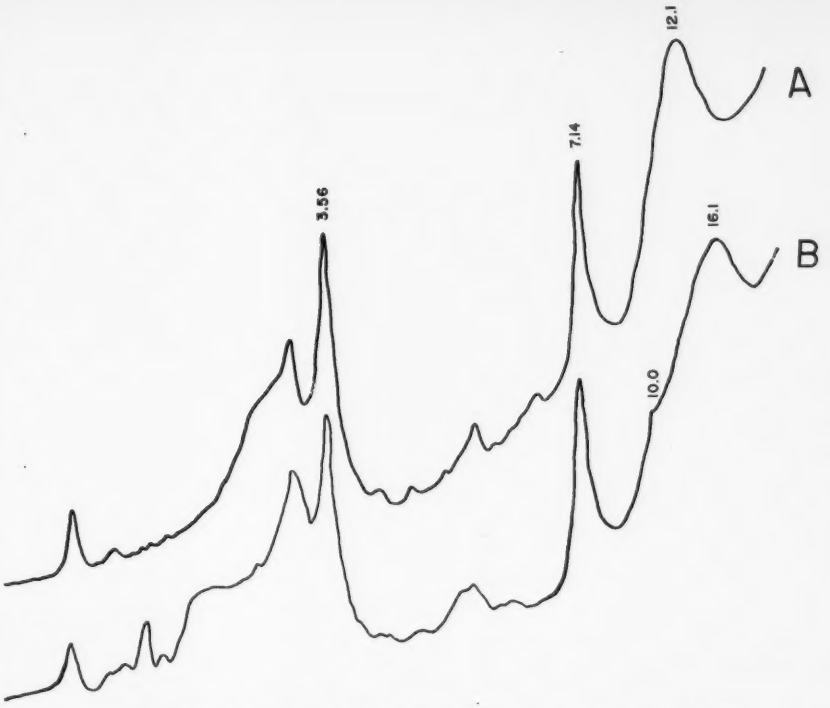

FIG. 5.—Diffraction patterns from oriented aggregates of a clay in equilibrium with 50% RH (a) and treated with ethylene glycol (b).

from an oriented aggregate at equilibrium with an atmosphere of 50% RH (a) and treated with ethylene glycol (b). The diffraction maximum in curve (a) for (001) occurs at 10.8 AU, suggesting from the Brown and MacEwan type curves shown along the legs of the four triangles in figure 6 that the sample contains a mixture of 75% 10 AU and 25% 15.4 AU minerals. The sample treated with ethylene glycol, curve (b), gives an apparent d-spacing of 11.5 AU, which suggests, from the "d' versus p" curve for mixture of 10 AU and 17 AU periodicities published by Weaver (1956) in his figure 2, that the mixture is of 75% 10 AU and 25% 17 AU. Both methods of analyzing this interlayed mixture of two periodicities give the same results, namely, 75% illite and 25% Ca-montmorillonite.

Figure 5 is a similar pair of diffraction patterns for another clay which could be described as being more poorly crystalline. The greater diffuseness, lower intensity and almost complete absence of higher orders of the non-kaolinitic part of the clay should be noted. Curve (a) is of the oriented aggregate in equilibrium with 50% RH and has a maximum intensity in the (001) region at 12.1 AU, which could be considered as a mixture of 10 AU and 12.4 AU material. The 12.1 AU maximum would then suggest, from the data on the 10 AU and 12.4 AU leg of the upper triangle in figure 6, that the mixture was of 80% Na-montmorillonite and 20% illite. On the other hand, if the mixture is considered to be of 10 AU and 15.4 AU material, an analysis of 40% Ca-montmorillonite and 60% illite would result.

Curve (b) is of the oriented aggregate solvated with ethylene glycol and shows a

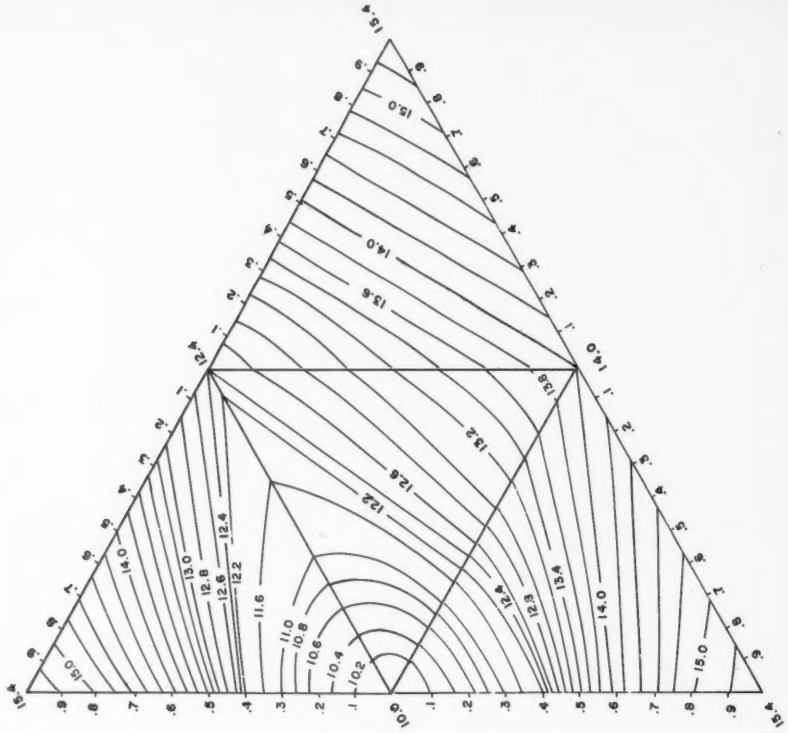

FIG. 6.—Three-component composition triangles for the four possible interlayer mixtures of 10 AU, 12.4 AU, 14 AU, and 15.4 AU clays. The apparent X-ray diffraction spacing, d', is contoured.

maximum at 16.1 AU. Consulting Weaver's (1956) "d' versus p" curve for mixtures of 10 AU and 17 AU periodicities, it could be concluded that the sample contained 60% montmorillonite and 40% illite. This analysis, unfortunately, agrees with neither of the possibilities suggested above by the information from curve (a). The sample was shown to contain no component diffracting at 14 AU. This was demonstrated by heating it to 300° C, which yielded a material that diffracted only at 10 AU. It could be tentatively concluded that the material which diffracted at 10 AU after the sample was heated had not been either all 12.4 AU or 15.4 AU montmorillonite, but a mixture of the two. The diffraction pattern in curve (a) is therefore produced from interlayer mixing of three different periodicities.

Hendricks and Teller's (1942) general equation could be adapted to consider three different periodicities. However, the resulting relationship would be too complex to be applied without the aid of an electronic computer; the theoretical curves would have to be corrected for variation in structure factor and combined Lorentz and polarization factors. These correlations are important in determining the position of the maximum intensities in curves like those shown in figure 2 for two periodicities. It is presumed that they would be even more important corrections for the three-periodicity curves. The impossibility of choosing a single acceptable set of correction factors for a mixture of three different clay minerals

seems to make the labor of calculating the theoretical curves useless.

A second, less rigorous method of interpreting diffraction from interlayer mixtures of three different clay mineral types is possible. Two-component curves of the three pairs of three different periodicities can be combined to form a triangular diagram. The triangular diagram represents mixture composition, and the vertical dimension which is contoured represents the d' or apparent diffraction periodicity for each composition. Figure 3 is a diagram of this kind for 12.4 AU, 14 AU, and 15.4 AU mixtures. The top triangle of figure 6 is a similar diagram for 10 AU, 12.4 AU, and 15.4 AU mixtures.

The 12.1 AU maximum from curve (a) of figure 5 would suggest a range of possible compositions represented by the 12.1 AU contour on figure 6. The quantity of 10 AU material in the mixture can be determined independently in the two-component 10 AU and 17 AU mixture produced by treating the sample with ethylene glycol. A unique composition in three components is determined by the intersection of the 12.1 AU contour line and the line representing 40% 10 AU material.

Interlayer mixtures of 14 AU illite and 12.4 AU and 15.4 AU montmorillonites can be treated in a similar manner. From the diagram in figure 6, which shows all four of the possible combinations of three clays, a range of possible compositions can be indicated by the contour line with the same value as the apparent spacing of the mixture in equilibrium with 50% RH. A unique composition is then determined by the intersection of the contour line with the percent chlorite line which can be evaluated from the two-component mixture of 10 AU and 14 AU resulting from heating the sample to 300° C, which dehydrates both Na- and Ca-montmorillonite allowing them both to diffract at 10 AU.

Mixtures of 10 AU illite, 12.4 AU montmorillonite, and 14 AU chlorite are analyzed from the (001) apparent spacing of the 50% RH sample in conjunction with the diagram in figure 6 to determine the possible composition range. The quantity of 14 AU chlorite is determined from the two-component mixture of 10 AU and 14 AU produced by heating the sample to 300° C to dehydrate the Na-montmorillonite. The intersection of the line of constant quantity of 14 AU material and the proper contour line represents the composition of the mixture in three components.

The same procedure is used to analyze interlayer mixtures of 10 AU illite, 14 AU chlorite, and 15.4 AU montmorillonite except that a different triangle of figure 6 is used. If the three components of the mixture are not clearly known from the experimental data, the several possibilities can be surveyed readily on this diagram, which includes all possible combinations of the four clay minerals that can enter into the interlayer type of mixing.

Occasionally the clay mineral that diffracts at 14 AU in the 50% RH sample will expand to 17 AU with ethylene glycol. This material has been called "swelling chlorite" by Stephens and MacEwan (1951) and later described by Grim and Johns (1953). When the swelling chlorite occurs in interlayer mixture with 12.4 AU and 15.4 AU montmorillonite, the glycol treatment becomes useless because it will not differentiate between the swelling chlorite and montmorillonite. Fortunately, this type of chlorite is not decomposed when heated to 300° C. Heat treatment can therefore be used to produce a two-component interlayer mixture of 10 AU collapsed montmorillonite and 14 AU chlorite. From the two-component mixture the quantity of 14 AU chlorite is determined. The three-component mixture is uniquely determined by the intersection of the chlorite quantity line and the apparent d' contour line.

The contour lines in the triangular diagrams are not determined or calculated; they are completely interpretative. The only calculated points on the contours lie at their intersections with the triangle, that is, where one of the three components has been reduced to zero. Errors introduced into the analysis of diffraction data because of poor interpretation are not likely to be significantly greater than the error in judging the apparent d' value of the maximum intensity on which the analysis is based. The results obtained from this type of analysis are expected to be reproduceable within one significant figure.

The method has been useful in studying large numbers of samples whose compositions, for easy comparison, must be characterized by numbers. Errors in the analyses are likely to be systematic and therefore unimportant in a comparative use of the data. If the same error is made in determining the composition of two samples, the results will still indicate, for example, that the right sample has the greater amount of montmorillonite. Regardless of its inaccuracy, the method provides a means of eliminating some of the personal judgment which often interferes with comparison of results obtained by two investigators.

The authors would like to acknowledge financial support from the Geology Foundation of The University of Texas which made this work possible.

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JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 29, NO. 1, PP. 87–97
FIGS. 1–2, MARCH, 1959

IDENTIFICATION OF CARBONATE MINERALS BY STAINING METHODS[1]

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ABSTRACT

The visual differentiation between the different mineral constituents in a carbonate rock is fundamental to most studies of carbonate petrography, correlation and genesis. Differential staining of the constituent minerals provides a rapid means of recognizing the textural and compositional differences in the rock. This contrast between the component minerals can be accentuated by etching prior to staining. For routine identification of carbonate minerals, alizarine red S and Harris' hematoxylin stains are recommended, since they are fast, efficient and dependable. For dolomitic rocks, the following twenty organic dyes provide a wide choice of colors for staining: Titan Yellow, 4(p-Nitrophenylazo)-1-naphthol, Quinalizarin, Alizarine Cyanine Green, Rhodamine "B" Base, Benzopurpin 4B, Congo Red, 1,5 Diphenylcarbolhydrazide, Alizarine Red S, Barium Eosinate, Trypan Blue, Safranine O, Anthraquinone Green G (and Green G Base), Janus Green B, Bismarck Brown Y, Carmine Cert., Orange G, Neutral Red, Hastings Light Fast Violet, and Celutate Brilliant Blue. A combination of two different stains, alizarine red S and Feigl's solution, can be used in differentiating dolomite, calcite, aragonite, high-magnesian calcite, gypsum, and anhydrite. Organic dyes stain calcite in acid solution and dolomite and magnesite in basic solution. The period of immersion in a staining solution, the acidity or alkalinity and temperature of the solution (i.e., whether the solution should be boiled or not) vary with the composition, porosity and grain size of the material to be studied. Because preparation and use of the solutions do not require laboratory facilities, the staining experiments may be conducted in any geological office on cores and cuttings for quick and accurate identification of these minerals.

Outlines of recommended staining procedures are given in tabular form.

INTRODUCTION

The geologist is frequently confronted with the problem of distinguishing between calcite, aragonite and dolomite (for compositions see table 1) in hand specimens or under the binocular or petrographic microscope. These carbonate minerals are difficult to distinguish because their physical characteristics are very similar. Yet the visual differentiation between calcite, aragonite, and dolomite is of fundamental importance to most studies of carbonate rock correlation and genesis. Differential staining of the constitutent minerals provides a means of establishing the identity of the carbonate minerals under study and to observe the textural and compositional relationship in carbonate rocks.

Staining methods have been used in carbonate analysis since Lemberg published his classical paper in 1887. The literature on staining techniques in the study of carbonate rocks has been reviewed by Rodgers (1940), Huegi (1945) and LeRoy (1950). The present study includes an up-to-date review and an experimental investigation with organic dyes that can be successfully adapted to carbonate rock analysis, particularly for quantitative mineralogical determinations under the petrographic microscope. The technique of staining analysis is also applied to new problems of carbonate differentiation, such as the identification of high- and low-magnesian calcite.

In this study the initial step was a review of the pertinent literature, followed by experimentation with the stains described, and an evaluation of their application to

TABLE 1.—*Composition of carbonate and sulfate minerals subjected to stain analysis*

Mineral	Composition
Calcite	$CaCO_3$ (Hexagonal)
Aragonite	$CaCO_3$ (Orthorhombic)
Dolomite	$CaMg(CO_3)_2$
Magnesite	$MgCO_3$
High-Magnesian Calcite	Solid solution of $MgCO_3$ in calcite
Gypsum	$CaSO_4 \cdot 2H_2O$
Anhydrite	$CaSO_4$

[1] Manuscript received July 30, 1958.

carbonate rock analysis. For the most part, the inorganic staining methods described in this paper have been taken from the literature. The organic stains, however, with few exceptions, either have not been previously used for carbonate analysis or the method of staining suggested here modifies existing techniques.

PROCEDURE

Treatment Prior to Staining.—All hand specimens are etched in dilute hydrochloric acid and washed in running water prior to staining. The acid solution consists of eight to ten parts by volume of commercial grade concentrated hydrochloric acid diluted with water to 100 parts as stipulated by Lamar (1950, p. 2) and Ives (1955, p. 8). Lamar (1950, p. 2) suggests five minutes etching time and Ives (1955, p. 8) stipulates eight minutes. In this study an etching period of two to three minutes was found to be adequate for most rocks. Experimentation soon shows the best etching time and acid concentration for a particular type of rock or purpose. The textural and mineralogical relationships of etched carbonate samples, particularly polished surfaces, appear in three dimensions under the binocular microscope.

Staining.—To obtain comparative results, each stain under consideration was tested at the same time and under the same conditions on calcite, dolomite, and calcite-dolomite intergrowths. For stains specific for aragonite, both calcite and aragonite were treated simultaneously, and for gypsum-specific stains, both gypsum and anhydrite were immersed in the staining solution at the same time.

The detailed method of preparation of stains specific for calcite, dolomite, magnesite, aragonite, high-magnesian calcite, and gypsum is given in the Appendix.

Treatment of Polished and Unpolished Surfaces after Staining.—Polished or unpolished surfaces of stained carbonate rocks after staining are mounted on glass slides with Canada balsam. For permanent storage they are covered with a cover glass which is held in position with sealing wax. This treatment cannot be extended to dolomite-specific stains which fade with time and have to be restored by immersion in dilute sodium hydroxide (NaOH).

Quantitative Mineralogical Analyses.—For quantitative mineralogical determinations of stained carbonates in thin section or polished surface under the petrographic microscope, the point counting technique of Chayes (1949) has been adopted.

RESULTS

Outlines of recommended staining procedures are presented in figures 1 and 2. For routine carbonate staining analysis a combination of two different stains: alizarine red S and Feigl's solution, can be used to differentiate between dolomite, calcite, aragonite, high-magnesian calcite, gypsum, and anhydrite (figure 1). Figure 2 provides an equally satisfactory scheme for staining analysis.

To follow the complete procedures outlined in figures 1 and 2, fresh chips can be used for each step or, using only one sample, the stained surface produced by one step can be rubbed off before the next test is applied. The concentration of appropriate acid or alkali is indicated below the rectangular box in each figure.

Three organic dyes, Harris' hematoxylin, alizarine red S, and trypan blue, were found to stain calcite. Table 2 indicates the color each of these dyes imparts to calcite.

Twenty-four organic dyes were found which can be employed in staining dolomite and magnesite of which twenty give excel-

TABLE 2.—*Organic stains for calcite*

Stain	Color Imparted to Calcite	Remarks	Supply House
Hematoxylin (Harris)	Purple	Excellent Stain	Hartman-Leddon Co., Philadelphia, Pa.
Alizarine Red S	Deep Red	Excellent Stain	Matheson Co., East Rutherford, N. J.
Trypan Blue	Blue	Poor Stain	Allied Chemical and Dye Company

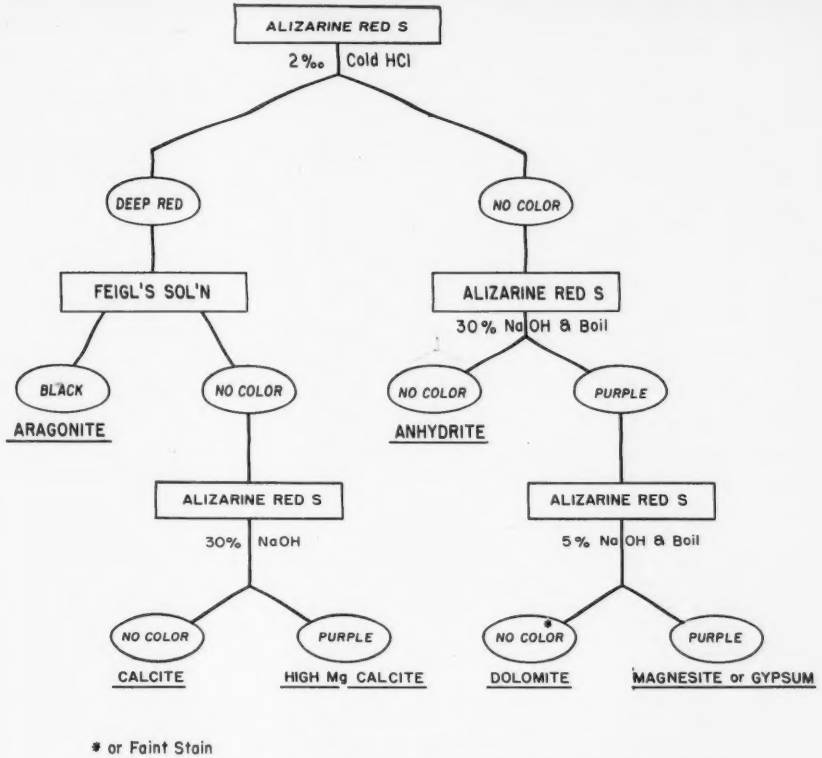


FIG. 1.—Recommended staining procedure I; alizarine red S and Feigl's solution.

lent results. Table 3 lists the dyes and indicates the color imparted to dolomite by each. It was found that magnesite will obtain a deep stain of the same color from these dyes in a much weaker alkaline solution than that needed to stain dolomite. Table 3 also lists the supply house from which each dye can be obtained. High-magnesian and low magnesian calcite can be differentiated with the aid of figures 1 and 2.

Table 4 lists four organic dyes which can be employed in staining gypsum.

Of the inorganic stains considered,[2] dyes specific for calcite include ferric chloride, copper nitrate and silver nitrate with potassium chromate. Hydrogen peroxide with caustic soda, potassium ferricyanide and ammonium sulfide with copper sulfate are specific for dolomite containing ferrous iron.

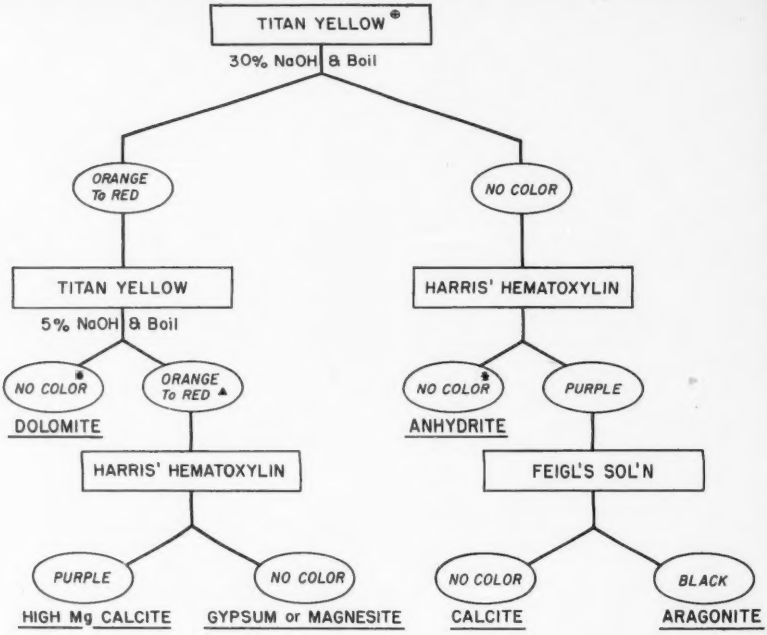
Cobalt nitrate (also known as Meigen's solution) and Ag_2SO_4 with $MnSO_4 \cdot 7H_2O$ (Feigl's solution) stain aragonite but do not affect calcite.

[2] The inorganic reagents mentioned in this paper can be obtained through all leading chemical supply houses, such as Merck, Mallinckrodt and others.

DISCUSSION OF RESULTS

Organic dyes which stain calcite but leave dolomite unaffected are active only in acid solution because of the greater solubility of calcite in cold dilute acid. The dyes which stain calcite in acid solution stain dolomite and magnesite in alkaline solution.

Alizarine red S and Harris' hematoxylin give the most satisfactory general-purpose



* Dolomite has a wide range of colors, any of the 20 excellent stains from Table 3 may be substituted for Titan Yellow to provide color contrast between dye & dolomite tested.

* Or faint stain (light orange)

▲ High-magnesian calcite used in this study was very fine grained. The behavior of coarse-grained high-mag. calcite was not studied.

FIG. 2.—Recommended staining procedure II; titan yellow, Harris' hematoxylin, and Feigl's solution.

stains of the organic dyes. They do not require boiling, and their action is fast, efficient, and dependable.

The use of hematoxylin is not new. Lemberg (1887) originally proposed the use of logwood, which contains the hematoxylin coloring substance. Steidtmann (1917) and Fairbanks (1925) modified the original Lemberg formula. However, some of the disadvantages of the stain were serious and were aptly summarized by Rodgers (1940, pp. 790–791) as follows:

"a. The stain is stable but the solution is not.
b. The stained coating contracts very strongly and tends to spall off if thick, therefore the best results are obtained with minimum immersion.
c. The stain rubs off very easily."

In the method described in this study (see Appendix), these difficulties have been overcome. The solution is stable, in fact it improves with time. After the solution is left overnight in an open beaker, its staining efficiency is increased, and the staining time per specimen can be reduced. Steidtmann (1917) suggested the use of a few drops of H_2O_2 to oxidize the dye, thereby increasing its staining power. However, in the present

TABLE 3.—*Organic dyes for staining dolomite and magnesite*[1]
(See Appendix for detailed method of preparation for stain)

Stain	Color Imparted to Dolomite	Remarks	Supply House
Titan Yellow	Deep orange red	Inadequate staining time gives yellow to yellow-orange	Eastman Organic Chemicals
4-(p-Nitrophenylazo)-1-Naphthol	Blue	Stain fades with time	Eastman Organic Chemicals
Quinalizarin	Blue		Eastman Organic Chemicals
Eosin Y	Deep Pink	Solution rapidly decomposes, thus poor dye	Matheson Company, East Rutherford, New Jersey
Alizarine Cyanine Green	Deep Green		Harshaw Chemical Company, Cleveland, Ohio
Rhodamine "B" Base	Purplish red		Allied Chemical and Dye Co.
Benzopurpin 4B	Pink		Eastman Organic Chemicals
1,5 Diphenylcarbolhydrazide	Deep Red		Eastman Organic Chemicals
Congo Red	Red		Allied Chemical and Dye Co.
Alizarine Red S	Purple		Matheson Co.
Barium Eosinate	Pink		Allied Chemical and Dye Co.
Trypan Blue	Blue		Allied Chemical and Dye Co.
Eosine Bluish	Yellow to Pale Orange	Poor Stain	Allied Chemical and Dye Co.
Safranine O	Reddish-pink		Matheson Co.
Anthraquinone Green G (and Green G Base)	Blue		Du Pont de Nemours and Co.
Janus Green B	Bluish-purple		Allied Chemical and Dye Co.
Hematoxylin	Purple	Poor Stain	Matheson Co.
Naphthol Green B	Yellow	Stain is too light	Allied Chemical and Dye Co.
Bismarck Brown Y	Orange-Brown		General Biological Supply House, Chicago, Illinois
Carmine Cert.	Purplish-red		General Biological Supply House, Chicago, Illinois
Orange G	Orange-red to red		General Billogical Supply House, Chicago, Illinois
Neutral Red	Orange		Matheson Co.
Celutate Brilliant Blue B	Blue		Harshaw Chemical Co.
Hastings Light Fast Violet (1 RS and 3 RL)	Violet		Harshaw Chemical Co.

[1] Magnesite will obtain a deep stain in a much weaker alkaline solution than that used for dolomite.

TABLE 4.—*Stains for gypsum*

Name of Stain	Color Imparted to Gypsum	Supply House
Rhodamine B Base	Purplish-red	Allied Chemical and Dye Co.
Barium Eosinate	Pinkish-red	Allied Chemical and Dye Co.
Titan Yellow	Orange	Eastman Organic Chemicals
Alizarine Cyanine Green	Bluish-green	Harshaw Chemical Co.

study, it was noted that H_2O_2 will decolorize the dye and decrease its staining effectiveness after a time, depending on the amount of H_2O_2 that was added. The specimens stained with Harris' hematoxylin, as proposed in this study, retain their even coat of purple color and do not peel or rub off readily when dry.

Thin sections and polished surfaces of artificial mixtures of calcite and dolomite stained with Harris' hematoxylin were subjected to quantitative mineralogical determinations under the petrographic microscope. Modal analyses agreed closely ($\pm 2\%$) with the actual composition of the artificial mixtures which indicates that this staining technique is satisfactory for quantitative mineralogical determinations under the microscope.

Rudi (1954) achieved satisfactory results in staining calcite with neutral red, methyl violet, methylene blue and fuchsin in acid solution. He prepared a 1 to 2% solution of the dye in distilled water acidified with glacial acetic acid (5 to 10 drops per liter). Magnesite and dolomite remained unaffected by these dyes. Hogberg (1950) used naphthol green and Hobbs (1954) employed Rhodamine B to stain calcite.

Ramsden (1954) used methyl red in an aluminum chloride solution to stain calcite. Mann (1955) stained dolomite by first treating a fresh rock surface with dilute hydrochloric acid. After vigorous action ceased, the same spot was treated with 1–2 drops of an alkaline solution of p-nitrobenzeneazoresorcinol. After 30 seconds, dolomite showed a blue color. The color is said to be dependent on the magnesium content.

Although there are many organic dyes (Welcher, 1948, p. 618) which can be used to detect magnesium, reference, in this paper, is made only to dyes which were found to stain dolomite effectively. In addition, several commonly used biological stains were found to stain dolomite satisfactorily. Logvinenko and Zabolotnaya (1954) and Piotrovskii (1956) recently reported on the use of some organic dyes in staining carbonate rock. Feigl and Leitmeier (1928) used diphenylcarbolhydrazide, one of the organic dyes recommended in this paper, for differentiating between dolomite and magnesite. Leitmeier and Feigl (1930) used p-nitrophenylazo-l-naphthol to distinguish calcite from dolomite, but found that the presence of the trace elements nickel, copper and cadmium interferes with the reaction.

Dolomite and magnesite are stained by organic dyes in alkaline solution only. When a solution containing Mg^{++} ions is made alkaline, $Mg(OH)_2$ precipitates and adsorbs the dye from solution, thus forming a colored precipitate. Dolomite is stained satisfactorily only in a boiling, concentrated (about 30%) caustic soda solution. Allowing the reaction to proceed without heat or decreasing the NaOH concentration increases the staining time. With Titan Yellow, for instance, a deep orange to red color is imparted to dolomite after about five minutes of boiling. If the reaction proceeds without heat, the staining will not be effective in less than half an hour to an hour.

For dolomitic rocks, 24 different organic dyes are available of which 20 give excellent results and provide a wide choice of colors for staining (Table 3). As the natural colors of dolomite range from white to red, pink, green, brown, gray and black, a stain that provides suitable contrast can be chosen. Thus for a pink dolomite, a green stain may be most effective, for green dolomite a pink stain, and for a white dolomite a blue stain provides the greatest contrast.

Silver nitrate with potassium chromate and ferric chloride are effective inorganic stains imparting a brown color to calcite.

The stains specific for aragonite (p. 95–96), high-magnesian calcite (p. 96), gypsum (p. 96) and dolomite containing ferrous iron

(p. 95) are satisfactory and provide rapid identification of the minerals.

In devising a staining method for a particular purpose, the period of immersion in the stain may have to be considered in terms of composition, porosity, and grain size. For example, the staining period of a polished surface of a fine-grained calcitic dolomite differs from that of a highly porous, unpolished aragonite-high magnesian-low magnesian calcite shell hash. The acidity or alkalinity of the staining solution is an important factor determining how effectively a specimen is stained.

The staining experiments can be conducted in any office for quick and accurate identification in working with hand samples, cuttings and cores. No equipment is necessary other than beakers and perhaps a porcelain evaporating dish and hot plate. Running water is desirable but not essential; the specimens can be washed in a large beaker if running water is not available.

CONCLUSIONS

1. For routine carbonate staining analysis a combination of two different stains: alizarine red S and Feigl's solution, can be used to differentiate between dolomite, calcite, aragonite, high-magnesian calcite, gypsum and anhydrite (figure 1).

2. Alizarine red S and Harris' hematoxylin are the most satisfactory organic general-purpose stains.

3. For dolomitic rocks, 24 different organic dyes are available of which 20 give excellent results and provide a wide choice of colors for staining (table 3).

4. Organic dyes which stain calcite but leave dolomite unaffected are active only in acid solution. Dolomite and magnesite are stained by organic dyes in alkaline solution only.

5. The stains for aragonite (p. 95–96), high-magnesian calcite (p. 96), gypsum (p. 96), and dolomite containing ferrous iron (p. 95) are satisfactory and provide rapid identification of the minerals.

6. In devising a staining method for a particular purpose, the period of immersion in the stain may have to be considered in terms of composition, porosity and grain size. The acidity or alkalinity of the staining solution is an important factor in determining how effectively a specimen is stained.

7. The staining experiments can be conducted in any office for quick and accurate identification with hand samples, cores or cuttings.

APPENDIX

A.—ORGANIC STAINS SPECIFIC FOR CALCITE

Harris' Hematoxylin (one of several types of alum hematoxylin solutions available).

Harris' hematoxylin can be purchased commercially or can be prepared as follows (Gurr, 1956, p. 419):

"Hematoxylin 10% in absolute alcohol	5	ml.
Mercuric oxide	0.25	gram
Potash alum 10% aqueous	100	ml.
Glacial acetic acid	4	ml.

Mix the hematoxylin and alum solutions; raise to boiling point then add the mercuric oxide and when the solution turns deep purple, turn off the heat; then cool and add the acetic acid."

The most satisfactory hematoxylin staining solution is made up of 50 cc. commercial grade Harris' hematoxylin and 3 cc. 10% hydrochloric acid.[3] The hematoxylin bottle should be well shaken before using. Three to ten minutes are required to stain polished and unpolished surfaces. The more frequently the solution is used, the quicker the stain takes effect. A fresh solution often requires as much as nine to ten minutes to stain a specimen. After the solution has been used frequently the staining period is reduced to three minutes and less. Calcite develops an even coat of purple color over its surface, dolomite remains unaffected.

Alizarine Red S

Dissolve 0.1 gram of alizarine red in 100 cc. 0.2% cold hydrochloric acid. (The hydrochloric acid solution is made up by adding 2 cc. of commercial grade concentrated hydrochloric acid to 998 cc. of water.) With this stain, calcite is stained deep red within two to three minutes and dolomite is not stained except on excess exposure. This for-

[3] A 10% hydrochloric acid solution is prepared by adding 10 cc commercial grade concentrated hydrochloric acid to 90 cc. of water.

mula is recommended in the staining procedure of figure 1.

An alternative, but not as effective, formula is made up as follows:

a. 2.5 grams of potash alum (Potassium aluminum sulfate, $KAl(SO_4)_2 \cdot 12H_2O$) is dissolved on heating in 400 cc of water. 50 cc of this solution is added, while hot, to
b. 0.25 grams of alizarine red dissolved in 5 to 8 cc of methanol.
c. 4 cc of 10% hydrochloric acid[3] is added to the above mixture.

In two to three minutes, calcite is stained deep red-brown, whereas dolomite is lightly colored, satisfactorily differentiating the two minerals.

Trypan Blue

Two formulas for trypan blue were developed. One formula consists of:

a. preparing an alum solution by heating and dissolving 10 grams of potash alum [$KAl(SO_4)_2 \cdot 12\ H_2O$] in 100 cc of water, and adding the hot solution to
b. 0.25 gram of trypan blue dissolved in 5 to 8 cc of methanol.
c. 8 cc of 10% hydrochloric acid[3] is added to the above mixture.

Calcite is stained deep blue with this solution, whereas dolomite is stained light blue, so that differentiation between the two minerals is possible.

An alternative and more easily prepared solution consists of dissolving 0.1 to 0.2 gram of trypan blue in 100 cc of water and adding 6 cc of 10% hydrochloric acid.[3] Both calcite and dolomite are stained, but calcite develops a deeper blue making it possible to differentiate between the two carbonates.

B.—ORGANIC STAINS SPECIFIC FOR DOLOMITE

The preparation of the solutions is the same for each dye listed in table 3: 0.2 gram of dye (for benzopurpin 20 cc is used) is dissolved in 25 cc methanol by heating, if necessary. Replenish any methanol lost by evaporation. Add 15 cc of 30% caustic soda solution[4] and bring the solution to a boil. Immerse the specimen in this boiling solution. The dolomite will be stained a specific color depending on the dye used after about five minutes' immersion (rarely it may take up to ten minutes). If the color of a specimen fades with time, it may be immersed in dilute NaOH (concentration is immaterial) and the original color will be restored.

C.—ORGANIC STAINS SPECIFIC FOR MAGNESITE

The preparation of the solution for each dye listed in table 3 is the same as for dolomite except that a 5% caustic soda solution[5] is used instead of a 30% concentration.

D.—INORGANIC STAINS SPECIFIC FOR CALCITE

Ferric Chloride

This stain was first used by Lemberg (1887, 1888, 1892) and other investigators have reported on it since (Keller and Moore, 1937, Rodgers, 1940).

Immerse the specimen in a 2½% ferric chloride solution[6] for a few seconds. The resulting stain imparts a brown color to calcite but does not affect dolomite. For greater contrast between calcite and dolomite, the Lemberg technique suggests thorough washing after staining with ferric chloride and immersion in an ammonium sulfide solution [$(NH_4)_2\ S + H_2O$][7] for a few seconds. Calcite will stain black, whereas dolomite remains unaffected. However, the black color will disappear on drying and the brown color obtained from the ferric chloride re-emerges.

Copper Nitrate

This method, suggested by Hinden (1903), Mahler (1906) and Spangenberg (1913) and modified by Rodgers (1940, pp. 797–798) consists of preparing a molar solution of $Cu(NO_3)_2$[8] in which the specimens are immersed for 2½ to 6 hours, depending on the intensity of coloration desired. When the specimens are removed from the nitrate solution, they should be immersed imme-

[4] A 30% caustic soda sclution is prepared by adding 70 cc of water to 30 grams of sodium hydroxide.

[5] A 5% caustic soda solution is prepared by adding 95 cc of water to 5 grams of sodium hydroxide.

[6] This solution is prepared by adding 97.5 cc of water to 2.5 grams of ferric chloride.

[7] This reagent is available commercially.

[8] The molar solution is prepared by adding 188 grams of $Cu(NO_3)_2$, 225 grams of $Cu(NO_3)_2 \cdot 3H_2O$, or 332 grams of $Cu(NO_3)_2 \cdot 6H_2O$ to 1000 grams of water.

diately in a strong solution of NH_4OH (concentrated commercial grade NH_4OH) for a few seconds, then washed and dried. Calcite is stained green or bluish-green, and dolomite is not affected.

Silver Chromate

The silver chromate staining method consists of immersing the specimens in a hot 10% silver nitrate solution[9] for two to three minutes, rinsing them in water, and immersing them in a saturated potassium chromate solution. Calcite obtains a chocolate brown stain, as do magnesite and gypsum, dolomite remains uncolored. This staining method is credited to Lemberg (1892) who devised various modifications of this stain, which are described in some detail by Rodgers (1940, pp. 791–792).

E.—INORGANIC STAINS SPECIFIC FOR DOLOMITE CONTAINING FERROUS IRON

Potassium Ferricyanide

A routine analytical test for iron can be applied if dolomite contains ferrous iron. Immerse the sample in dilute hydrochloric acid (1:50–100) containing a few drops of potassium ferricyanide. Dolomite will stain deep blue, whereas calcite will not be affected. This test was originally proposed by Heeger (1913).

Hydrogen Peroxide and Caustic Soda

Dolomite may be distinguished from ankerite (ferriferrous dolomite) by treatment with hydrogen peroxide and caustic soda. Ankerite takes on an orange color, whereas pure dolomite does not (Hallimond, 1925, p. 39). According to Taylor (1948, p. 33), the depth of the stain is related to the proportion of ferrous carbonate present. This experiment was not repeated in the current study and therefore more detailed directions for the preparation of these solutions are not given. The effect of a large amount of replacement of iron by manganese in ankerite was not determined.

Ammonium Sulfide and Copper Sulfate

Kirchberg (1940, 1941) developed the following method for staining ankerite: A polished surface of the sample is etched for two minutes in 10% hydrofluoric acid,[10] washed thoroughly in running water and dried with a cloth or paper towel. It is subsequently immersed for one minute in an ammonium sulfide solution,[11] washed in water and dried. This treatment is followed by immersion for five minutes in 10% copper sulfate solution[12] and after washing in water and drying it is placed again in the ammonium sulfide solution. If necessary, this treatment may be repeated several times. Ankerite takes on a gray-black color.

F.—STAINS SPECIFIC FOR ARAGONITE

Feigl's Solution

A sensitive test, described by Feigl (1937, pp. 328–329), depends on the slightly different solubilities of aragonite and calcite in water. The method of preparing the reagent (Feigl's solution) is described by Feigl (p. 329), as follows:

> "Solid Ag_2SO_4[13] is placed in a solution of 11.8 grams $MnSO_4 \cdot 7H_2O$ in 100 cc of water, and boiled. On cooling, the mixture is filtered and one to two drops of dilute NaOH solution are added and the precipitate which forms is filtered off after one to two hours. The solution should be kept in dark bottles."

Aragonite stains black in this solution, whereas calcite and dolomite remain unstained.

An alternative test for distinguishing between aragonite and calcite consists of boiling the sample in cobalt nitrate solution (Meigen's reaction, Holmes, 1921, p. 262). According to Togari and Togari (1955, p. 57) 2 cc. of 0.1 N $Co(NO_3)_2$ solution are added to 0.2 g of sample, brought to boiling and filtered. Williams, Turner and Gilbert (1954) state that boiling for about twenty minutes is necessary. Aragonite assumes a lilac purple color, whereas calcite remains unaffected. However, Jirová (1956) reports that he applied Meigen's solution to the tests of three species of foraminifera. The

[9] The silver nitrate solution is prepared by adding 90 cc of water to 10 grams of silver nitrate.

[10] This solution of hydrofluoric acid is prepared by adding 20 cc of commercial grade 48% hydrofluoric acid to 80 cc of water.

[11] This reagent is available commercially.

[12] Ten grams of copper sulfate in 90 cc of water.

[13] This reagent is available commercially.

reaction indicated aragonite, but x-ray powder patterns were that of calcite.

G.—STAINS SPECIFIC FOR HIGH-MAGNESIAN CALCITE

An alcyonarian coral, *Plexaurella kunzei* of Recent age from Florida, was used in this experiment as a good sample of high-magnesian calcite. This material is made up of 100% high-magnesian calcite and has a $MgCO_3$ content of 11.2%. The dried sample has a brownish color, but treatment with ordinary commercial Clorox renders a white powder.

The high-magnesian calcite stains with Harris' hematoxylin like any calcite, i.e., the stain imparts a purple color to the mineral. However, to Titan Yellow, Alizarine Red S and Alizarine Cyanine Green, high-magnesian calcite reacts in the same manner as dolomite. In the staining experiment the spicules of *P. kunzei* were stained a deep yellow with Titan Yellow, whereas the rectangular particles of the organism assumed an orange color. It was assumed that the degree of coloration reflects the amount of magnesium present in the material. This assumption was verified by subjecting the different stained particles to spectrographic analysis. The spicules which stained yellow had a Mg/Ca ratio of 0.126. The rectangular particles which stained orange had a Mg/Ca ratio of 0.482.

H.—STAINS SPECIFIC FOR GYPSUM

Dissolve 0.1 to 0.2 grams of any of the four dyes specific for gypsum (see table 4) in 25 cc of methanol. Add 50 cc. of 5% NaOH.[5] Immerse the specimen in the cold solution. Staining takes place within a few minutes, imparting a deep color to the gypsum and a very faint tint of the same color to the dolomite. The color difference is sufficient to distinguish the two minerals easily. Heating the solution increases the staining effectiveness. Anhydrite and calcite are not stained by these dyes.

Mégnien (1957, p. 29) reports on the use of a 10% mercuric nitrate solution acidified by 1% nitric acid which imparts a lemon-yellow color to gypsum. An alternative method suggested by Mégnien consists of heating gypsum to a temperature above 102° C over a Bunsen burner when gypsum is converted to $CaSO_4 \cdot \frac{1}{2} H_2O$ resulting in a brilliant white coloration of the mineral.

I.—STAINS SPECIFIC FOR ANHYDRITE

No effective stains were developed for anhydrite. The presence of anhydrite is determined by the process of elimination as indicated in figures 1 and 2.

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JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 29, NO. 1, PP. 98–103
FIGS. 1–6, MARCH, 1959

A PHOTOMULTIPLIER PHOTOMETER FOR STUDYING QUARTZ GRAIN ORIENTATION[1]

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ABSTRACT

In this article an improved photometer for studying quartz grain orientation is described. The nonlinear illumination effects caused by a phototube having a cylindrical cathode and an axial wire anode have been eliminated in this new system by the use of an infrared sensitive photomultiplier tube with a flat photocathode. Because of this improvement, the preferential quartz grain orientation of many thin sections which could not be measured before can now be determined.

INTRODUCTION

The photometer method for determining the direction of quartz grain orientation described by Martinez (1958, p. 588) basically consists of measuring by means of an attached photometer the variation in the intensity of monochromatic light passed through a standard thin section of sandstone on the stage of a petrographic microscope, with gypsum plate inserted and nicols crossed, during a 360° rotation of the stage. A light intensity minimum occurs when the trend of the optic axes of the quartz grains lies parallel with the slow direction of vibration of light in the gypsum plate. Experimental data interpreted by Martinez indicate that the trend of the long axes of the quartz grains also lies parallel with this direction in some samples.

In the photometer method for determining the direction of quartz grain orientation described by Martinez, the photometer light detector is a vacuum phototube consisting of a half-cylinder metal cathode and an axial wire anode. Since the cathode is curved and the wire anode casts a shadow, the direction sensitivity of the phototube is not uniform. Hence, if the light pattern from the sandstone thin section is nonhomogeneous, an accurate measurement of the optical characteristics of the quartz grain fabric may not be possible. If a phototube with a flat cathode could be used, the nonlinear effects caused by the geometry of the phototube originally used would be eliminated.

[1] Manuscript received August 6, 1958.

APPARATUS

Flat cathode phototubes of the photomultiplier type are used in scintillation counter applications; hence a type C-7160 photomultiplier tube with an S-1 type spectral response peaking at approximately 8000 Angstrom units was obtained from the Radio Corporation of America. A suitable mounting housing was fabricated for the photomultiplier tube, and the tube was wired into a modified bridge circuit with biasing voltage and an indicating galvanometer. Figure 1 is a picture of the complete system which displays the photomultiplier-tube housing, the petrographic microscope, the junction and control box, and the Rubicon galvanometer.

Photomultiplier-Tube Housing

The photomultiplier-tube housing extending from the top of the petrographic microscope can be observed in figure 1. An inverted, U-shaped cable clamp is attached to the top of the housing and restricts the cable movement at the entrance to the housing so that cable bending will not cause internal electrical short circuits. Figure 2 shows the housing itself, and figure 3 shows a cross-sectional view. The housing is aluminum, and the tube's magnetic shield is "nicolai" or mu metal and is grounded. A plastic mounting ring fastens the shield to the tube. The tube is protected from shock at the light entrance by cork and felt gaskets; the tube is plugged into a plastic tube base which is mounted on the aluminum housing shelf.

The housing circuit containing the photomultiplier tube can be seen in figure 4. The

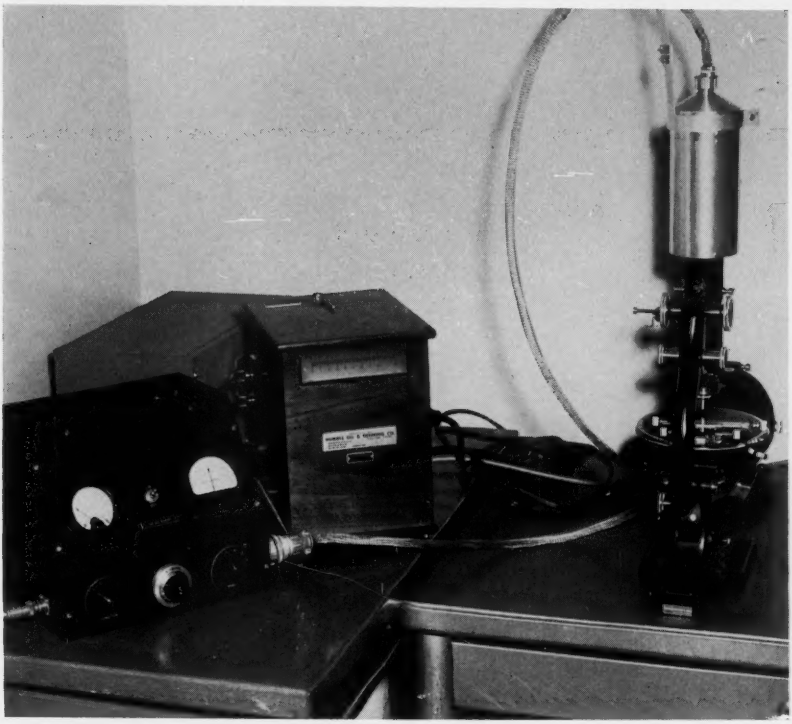

FIG. 1.—Complete system.

tube voltage-divider resistors and cable ends are soldered to connections on the tube base. A wire whose insulation can withstand several thousand volts connects the semi-transparent photo cathode pin to a negative high-voltage source of approximately 1200 volts. Each successive dynode is connected to a higher positive potential along the voltage-divider chain. The positive high voltage is connected to ground. It can be seen that the mu metal shield and the cable shield are connected to ground. The signal comes out on the anode lead.

Junction and Control Box

Figure 5 is a photograph of the junction and control box. The box houses meters, controls, and a bias battery and is a distribution junction for voltages and the signal. The bias battery is mounted inside the box toward the back, and the meters and controls are mounted on the front panel. There are cable receptacles on each side of the box. A high-voltage cable receptacle is located on the left-hand side, and the photomultiplier-cable receptacle and galvanometer binding posts are located on the right. The zero-to-fifty microampere meter mounted in the upper left of the front panel gives a current reading proportional to the absolute value of the light intensity. Located in the upper right-hand corner of the front panel is a twenty-five-to-zero-to-twenty-five microampere galvanometer used for biasing and coarse balancing. The galvanometer shorting switch between the two meters can be used to connect a short across the galvanometer binding posts for any position of the function switch. In the lower left-hand corner of the front panel is the function switch which

FIG. 2.—Photomultiplier-tube housing.

has four positions: Position one is the "off" position; position two gives a light-intensity reading on the zero-to-fifty microampere meter; position three is used to obtain a coarse galvanometer balance by means of the bias adjustment; and position four is the sensitive galvanometer position where the actual grain orientation readings are taken. To the right of the function switch is the bias adjustment knob; and to the right of the bias balance adjustment knob is the galvanometer current range switch. Position one gives the largest galvanometer current, and each larger numbered position cuts the current of the preceding position in half. Figure 6 is a circuit diagram of the internal wiring of the junction and control box and gives information on some of the circuit components. The sensitive galvanometer is a product of the Rubicon Company of Philadelphia and has specifications as follows:

Type Number	3413
Sensitivity	0.004 μa/mm
Resistance	452 ohms
Period	2.9 seconds
C. D. Rx	5000 ohms

The function switch is a Mallory rotary type with four decks and four positions, or equivalent. A shielded cable with a well insulated high-voltage lead connects the junction and control box to the photomultiplier-tube housing.

High-Voltage Power Supply

The high voltage is obtained from a "Model 312 Super-Stable High-Voltage Supply" made by Baird-Atomic Incorporated of Cambridge, Massachusetts. The regulation of this power supply is sufficient to minimize small erratic voltage excursions and voltage drift which produce objectionable changes in the galvanometer light-spot position. For the grain orientation system, this power supply is operated in the negative high-voltage position at approximately 1200 volts.

OPERATION

In placing the grain orientation system in operation, one might proceed with the fol-

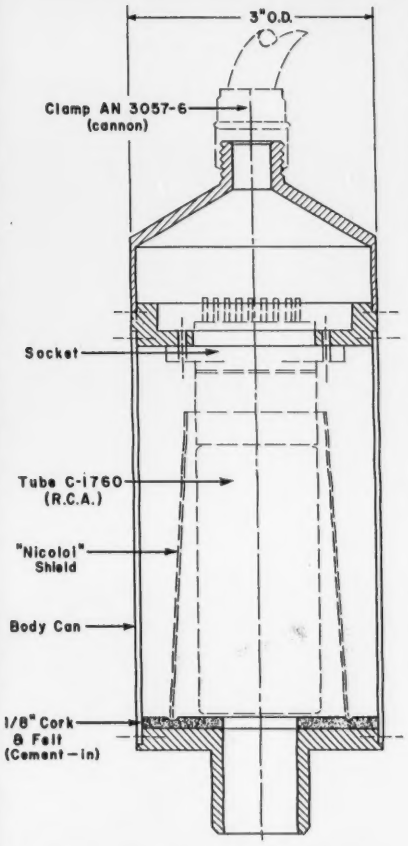

FIG. 3.—Cross-sectional view of photomultiplier-tube housing.

lowing sequence of operations. Connect the system together electrically with the proper cables. Turn the function switch to position one and the galvanometer shorting switch to short. On the high-voltage power supply, select negative high-voltage operation and turn the voltage switch to position "M." Close the high-voltage supply power switch. Also, switch on the sensitive Rubicon galvanometer and the sample-illuminating light. While the system is warming up for several minutes, one can focus the petrographic microscope on a desired field of vision of the thin section to be examined. After warm-up, replace the eyepiece of the microscope with the photomultiplier housing and turn the function switch to position two and read the maximum light-intensity current value. Now, turn the function switch to position three, select a safe Rubicon galvanometer current operating range by observing the coarse galvanometer on the junction box while rotating the microscope stage and by setting the galvanometer current range switch, and balance the galvanometer using the bias balance adjustment knob. Turn the function switch to position four, turn the galvanometer shorting switch to open, and made a fine bias balance adjustment while watching the spot indication on the sensitive Rubicon galvanometer. At this time, it may be necessary to reset the current range switch. The system is now in operation and grain orientation measurements as described by Martinez can be made. To cease operations, one should short the galvanometer, turn the function switch to position one, switch off the high-voltage supply, switch off the Rubicon galvanometer, and switch off the sample-illuminating light.

CONCLUSIONS

When placed in operation, it was found that the amplification of the photomultiplier tube was sufficient so that the use of the vacuum-tube amplifier used initially could be discontinued; and the nonlinear effects were considerably reduced. Improvement in the results of grain orientation observations was such that the photomultiplier system was pressed into immediate use. The preferential quartz grain orientation of many thin sections which could not be measured before can now be determined.

ACKNOWLEDGEMENTS

The writer wishes to express his appreciation to the Humble Oil & Refining Company for permission to publish this information and to certain of his colleagues who furnished valuable assistance. J. D. Martinez gave helpful advice and encouragement during the building of this equipment, and Edwin Statham helped considerably during the initial testing period.

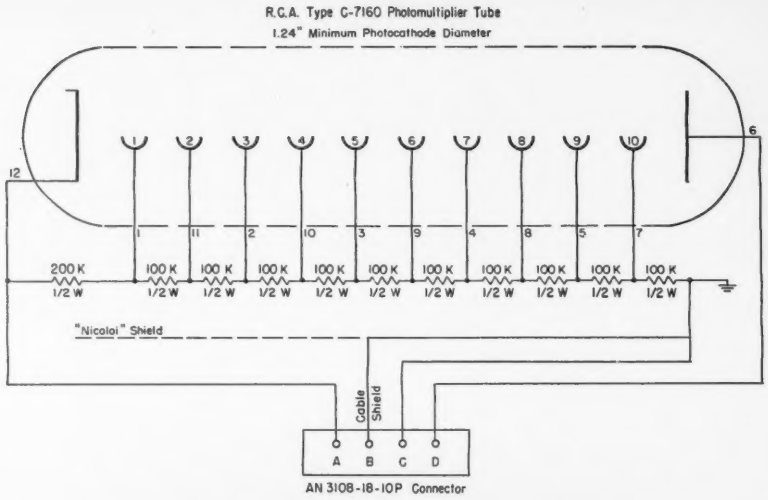

FIG. 4.—Photomultiplier-tube housing circuit.

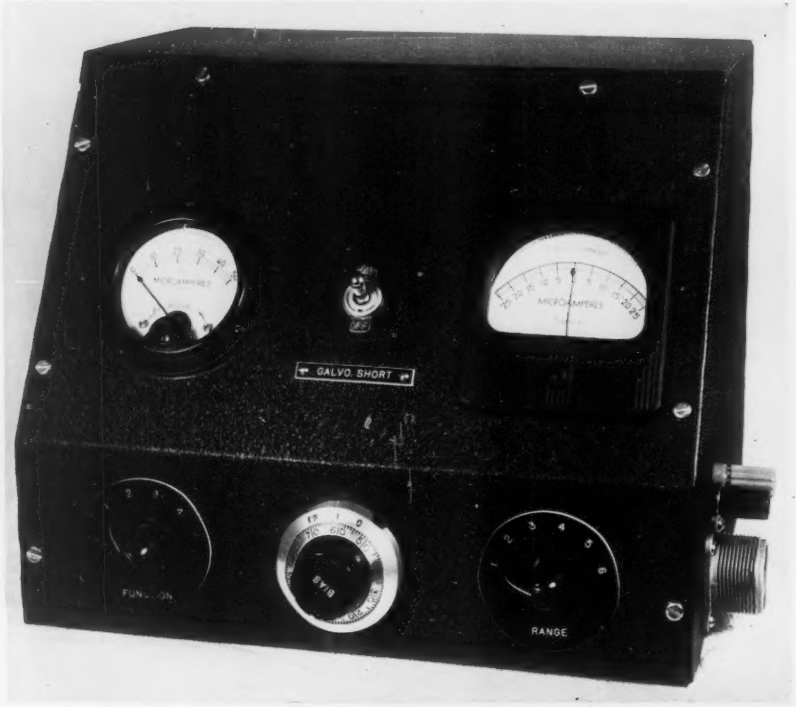

FIG. 5.—Junction and control box.

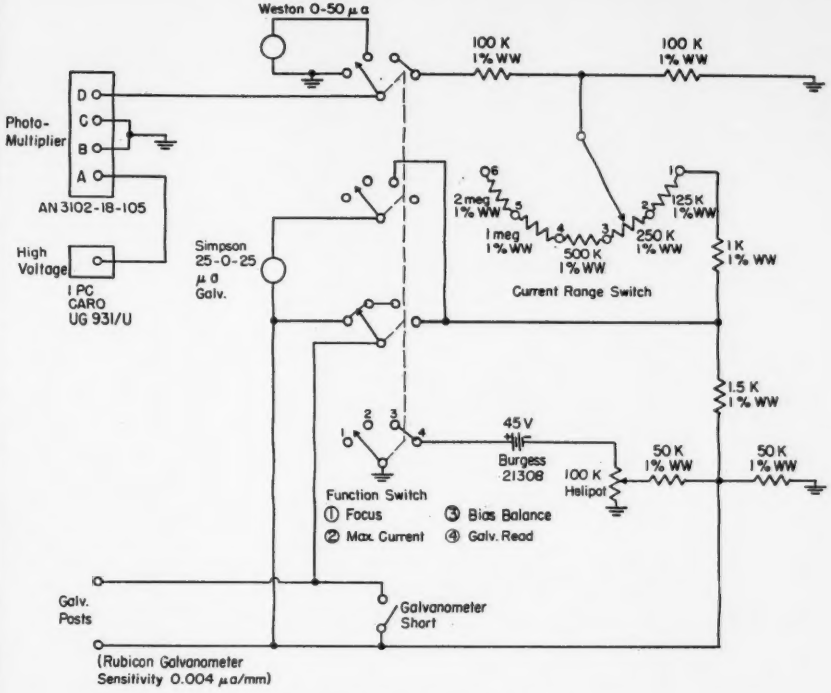

FIG. 6.—Junction and control box circuit.

REFERENCE

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JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 29, NO. 1, PP. 104–107
PLATE 1, MARCH, 1959

NEW METHOD FOR STUDY OF CALCI-PHOSPHATIC PELLETS[1]

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ABSTRACT

Hydrofluoric acid treatment of opaque calci-phosphatic pellets renders them translucent. Details of internal structures, such as size, composition, configuration of nucleus, and number and thickness of growth bands, can readily be studied in any number of specimens. This facilitates comparison by horizon and locality, of genesis and growth of pellets, bottom conditions, and paleoecology in large pellet-populations.

INTRODUCTION

A series of papers on the conversion of calcite to fluorite (Grayson, 1956; Sohn, 1956; Upshaw, et al., 1957) led the writer to experiment with the possible fluoridation of calci-phosphate pellets by hydrofluoric acid treatment. Further impetus for this study was derived from the author's recent work on conodont-controlled pellets (Tasch, 1958). In the latter study it was noted that the residue consisted of phosphatic films of the collophane type when such pellets were placed in HCl. The carbonate fraction was released as carbon dioxide gas.

Prior to the findings reported here, individual light-to-dark gray pellets a few tenths of a millimeter in longest diameter were mounted on glass slides and ground down by hand. Since, under reflected light the pellets are dense opaque objects from which little could be gleaned other than the nature of the surface and the general shape (fig. 1), the use of thin sections was, of course, indicated. Data were needed on the following characteristics of these pellets: variety of mineral and fossil material serving as nuclei, configuration and size of nuclei compared with that of the pellets and mode of growth. However, thin sectioning was very time consuming, and was a precarious operation in the final stages of grinding, since portions of the remaining slice were often lost in the process.

[1] This research was supported in part by two grants from the National Science Foundation (G-764, G-2933). A University of Wichita Research Grant financed the preparation of this paper. Manuscript received June 3, 1958.

PROCEDURE, REACTIONS, AND CRYSTAL CHEMISTRY

The procedure in fluoridizing the pellets was as follows: small quantities of dried but otherwise untreated pelletiferous shale samples were poured into polyethylene containers. Each sample was then covered with a commercially prepared 48 percent solution of hydrofluoric acid and allowed to stand several minutes.

The action was vigorous, resulting in the generation of a considerable amount of heat and the liberation of a gas. When this gas was passed through limewater, a white calcium carbonate precipitate formed—hence the gas was carbon dioxide. This confirmed previous observations that the pellets were both carbonate and phosphate.

Grayson (1956, p. 71) reported the following end products for the HF reaction with carbonate: CaF_2, CO_2 and water. Without detailed chemical and X-ray evidence for the HF reaction with the calci-phosphatic pellets of the present study, one can but speculate that fluorapatite [$Ca_{10}F_2$ $(PO_4)_6$] may also have formed. This speculation may be supported by the following observations: in naturally occurring phosphate rock, fluorapatite is reported to be "the essential constituent"; tricalcium phosphate has proven efficacious in removal of fluorides from potable water by forming fluorapatite (Mansfield, 1940, p. 865, 872).

When the reaction had ceased, the acid was poured off and the sample was washed into a petri dish and covered with a thin layer of water. The contents of the petri dish were then observed under transmitted light using an ordinary binocular micro-

scope. Even at low magnifications the pellets appeared translucent. However, all phosphatic and calcitic objects which were not too thick, appeared translucent and showed details of internal structure at magnifications of × 100 or higher.

After standing for several days under a thin layer of water, many of the translucent pellets darkened and tended to become opaque. While this observation does not extend to all pellets in a given sample, it suggests that some unstable intermediate compound may have formed and slowly converted to the stable condition.

In Grayson's experiment (op. cit.) it seems reasonable to attribute the change from opaque to translucent to the removal of the CO_3 radical and substitution of F atoms surrounding each Ca atom (cf. Niggli, 1954, fig. 55). However, a more complex explanation seems indicated in the reaction observed by the writer, since phosphate as well as carbonate was involved. Resolution of the matter must await interpretation by some expert since the mineralogy and chemistry of apatites is extremely complex and fine points are still in debate (Trautz, 1955, p. 698–702).

OBSERVATIONS

The detailed structures of hundreds of calci-phosphatic pellets were readily observable as different portions of the sample were scanned under the microscope. The size of the respective nuclei, the mineral or fossil nature of nuclei, the thickness of successive increments, usually concentric bands, could be determined in a manner of minutes for any sample (fig. 2).

Pellets after fluoridation were golden yellow-to-amber colored. In many instances distinct color bands of deep red or orange that contrasted strikingly with previous or subsequent bands in the same pellet were observed against the amber background. These were iron oxides that stood out against the uniform background color.

Where nuclei were found to be opaque, they were observed under reflected light to determine the nature of the opaque material. Relatively thick fragments of shell material, even though fluoridized, will remain opaque. Dense organic material and opaque minerals account for other opaque nuclei. It should be noted that the pellet growth about such opaque nuclei appeared translucent in transmitted light. Many nuclei appeared translucent.

Despite the fact that most nuclei were irregular and sharp-edged, the resulting pellet was always ovate or elliptical. This indicated that the mode of growth was regulated by some invariable or slightly variable control. Such a control could have been the relative stability of a sedimentary particle adhering to any given point, as contrasted with any other, on an irregularly shaped, minute, rolling object (fossil or mineral). In numerous instances, the pellet was almost all nucleus. Thus, many pellets with a maximum diameter of 0.4 mm were composed of a comparatively large, irregularly shaped shell fragment around which a slight accretion of sediment smoothed out the irregularities and ensured the ovate or elliptical configuration.

Generation of CO_2 caused mechanical breakage and radial fracturing of many pellets. This is seen in several pellets where the mineral grain constituting the nucleus had been freed from the concentric bands enclosing it on one end only. These bands are often seen in an unfurled condition. The radial fracturing suggests that the gas was largely released from calcitic shell fragments forming many of the pellet nuclei. A few cases were observed where, in the early history of the pellet, fracturing occurred, and subsequently the fractured initial pellet had been enclosed by added concentric bands. Primary structures of this type can be distinguished from those due to fluoridation by the fact that in the latter, radial fractures reach the periphery of the pellet.

Since the samples studied contained an abundant microfauna as well as pellets, and since most of these were also calci-phosphatic, delicate and fine structures of ostracods, brachiopods, molluscs, etc. as well as the internal structures of pellets, were observed in the prepared material.

APPLICATIONS

A more exhaustive report on this subject is in preparation. Utilizing the method described in this paper, the writer is now studying pellet-populations and their changes laterally and vertically. The bearing that a study of this type can have on problems such

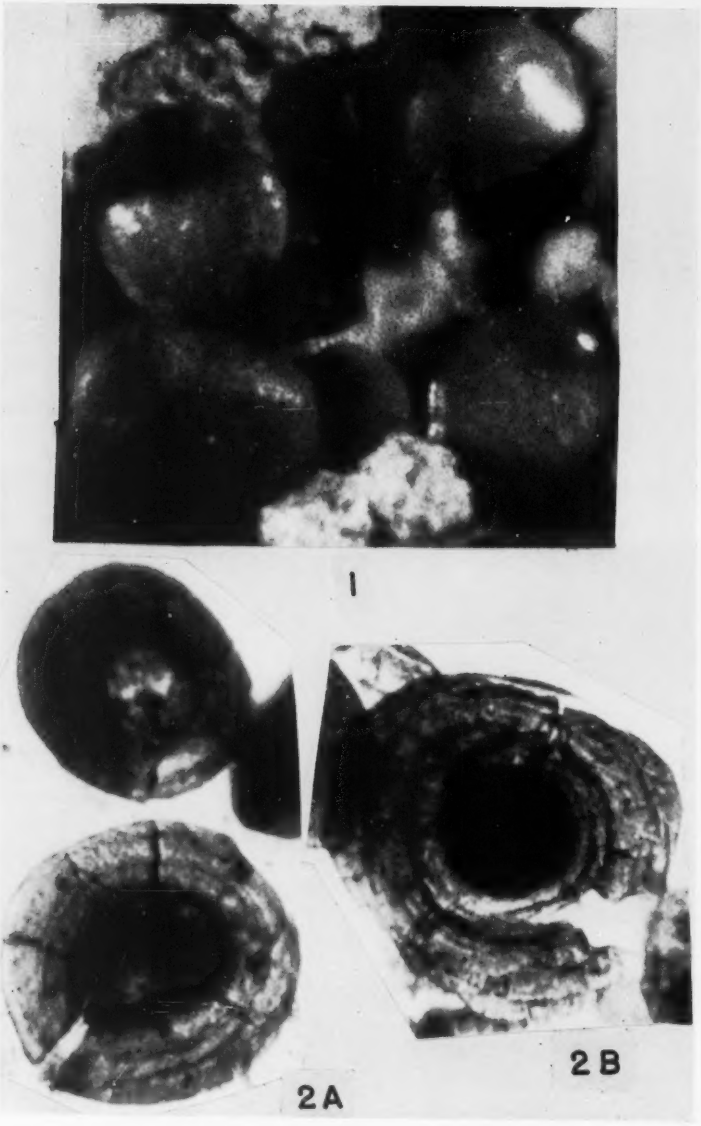

PLATE 1

as the sedimentary history of a formation, details of bottom conditions, paleoecology, and the index-value of pellets in stratigraphy, while still to be demonstrated, is sufficiently promising to encourage much research in this area.

ADDENDUM

Professor W. D. Keller has kindly run some diffraction analyses on two samples I submitted. The diffractograms are in my files and available to anyone interested. Sample 1 consisted of commercially prepared tricalcium phosphate that had been treated with a 48 percent solution of hydrofluoric acid. The diffractogram indicated fluorite. Keller wrote that "if any fluorapatite is present it must be in quite small quantities."

Basal Maquoketa pellets gave a canary-yellow precipitate when we ran it through the ammonium molybdate test. The presence of phosphorus was thus indicated. Fluoridized pellets run through the same test yielded no precipitate. Sample 2 consisted of fluoridized pellets. The diffractogram indicated fluorite. Keller observed that it was "probably poorly crystallized."

These results indicate that relative to calcium carbonate the phosphate content of the pellets is small; that fluoridization may have a masking effect on the phosphate. The presence of fluorapatite in these two samples was not indicated by diffraction analysis. For the Maquoketa pellets the explanation may be the original relatively small quantity of phosphorus present.

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EXPLANATION OF PLATE 1

FIG. 1.—Calcium-phosphate pellets of the basal Maquoketa, loc. 6, Sperry Twp., Clayton Co., Iowa. Reflected light. ×150.

FIG. 2A.—Fluoridized pellets photographed by transmitted light. Note that these pellets though appearing as thin sections, have the same configuration and volume as pellets in Fig. 1 with the additional feature of translucence. The nucleus is semiopaque. Seven concentric bands can be counted. The radial fractures and separation of the pellet from its nucleus in the lower left sector are due to the escape of carbon dioxide during the fluoridation reaction (cf. fig. 2B). Upper pellet has a translucent nucleus and shows the same features as fig. 2A but it is out of focus. ×150.

FIG. 2B.—Nine concentric bands of calcium phosphate formed about a rounded opaque nucleus. Note variation in thickness of bands. Hundreds of determinations of this kind for any sample can be made in a matter of minutes and genesis of pellets by horizon and locality compared. ×150.

JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 29, NO. 1, PP. 108–115
FIGS. 1–2, MARCH, 1959

THE SLOTTED CONE SPLITTER[1]

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ABSTRACT

A laboratory sample splitter was designed in the form of a 60° cone over whose surface the entire sample passes. The split is captured by falling through pie-shaped slots on the surface of the cone. The splitter sampled an average of 13.5 percent of a crushed quartz monzonite, of which 80 percent passes a 140-mesh sieve.

The amount of sample captured is dependent on the particle size of the material sampled. A line of regression of the percent captured on size and confidence limits for the estimate of the means of the amount split are shown. Some qualities desirable in a splitter are enumerated and the conformity of several splitters to these criteria is discussed.

INTRODUCTION

Sampling is an integral part of most physical sciences and especially in geological and geochemical problems. The geologist has the problem of sampling a large mass of rock, and from this about 100 grams must be subsampled for chemical analysis. The chemist must split off a still smaller sample for chemical analysis.

The literature on sample splitters covers the range from a "thief" for sampling carloads of crushed rock to the microsplit (Otto, 1933) for laboratory sampling, and from quartering by hand to the mechanical sampling in ore milling operations. The amount of material sampled may range from extremely large, as in a thief in the hold of a ship, to approximately twice the number of grains necessary for grain counting when the microsplit is used. The sampling may be done with a single component or multicomponent material which can be present in one or more sizes.

Because of the wide range of requirements, some desirable qualities in a laboratory splitter can be enumerated:

1. The splitter should be able to reduce both small and large amounts of sample.
2. The splitter should be capable of sampling a wide variety of particle sizes.
3. The time required for the operation should be short.
4. All particles in the lot to be sampled should have an equal chance of being sampled.
5. The sampler should be easily cleaned and require a minimum of maintenance.
6. The operation of the sampler should be simple.
7. The materials of which the splitter is made should not contaminate the sample.

Some forms of laboratory samplers can be considered in terms of their conformity to the attributes outlined above. Splitters of the Jones type including the smaller microsplit, which reduce a sample by sending alternating halves of the sample to opposite pans, require a longer time for splitting than some other types. They have the added disadvantage that particles might lodge in small apertures that may exist between the separators and the triangular chutes which alternate in direction. A serious bias in trace element analysis could be introduced if the particles lodged in the gaps were the only ones containing the trace element of interest. These gaps could be filled by soldering, but in some laboratories, where emphasis is on lead age determinations, exposure of samples to soldered parts is extremely undesirable. Similar precautions should be observed for other components of solders. However, if splitters were made without soldered joints, such precautions would be unnecessary.

McKinney and Silver (1956) have recently improved the qualities of the micro-

[1] Publication authorized by the Director, U. S. Geological Survey. Manuscript received July 20, 1958.

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split. The three-dimensional geometry of the splitter is laid out in two dimensions on a single piece of sheet metal, and by means of appropriate bends and folds the splitter is formed without joints.

Wentworth and others (1934) devised a splitter in which the sample is allowed to pour onto a revolving horizontal plate. Around the periphery of the plate are several sample bottles which allow the operator to vary the size of the split from the original sample. Kellagher and Flanagan (1956) have described a multiple cone sample splitter designed for grain counts. The size of the split may be varied but the splitter does not work satisfactorily for powders. Krumbein (Otto, 1933) used the principle of two knife edges intersecting at right angles to design a splitter for quartering small samples for grain counting.

DESIGN OF THE SPLITTER

A cone would be a good geometric figure on which to base a sampler because the surface of a cone has radial symmetry. Either powders or grains could slide down the surface of the cone, and inasmuch as the whole sample would be split, each particle would have an equal chance of being captured. In place of the pie-shaped pans used in the multiple cone splitter, slots of a similar area can be cut into the face of the cone and the sample would fall through the slots into a container.

The slots can be made as small as desired to minimize the time required for splitting. Although two or more slots can be symmetrically arranged, four slots, ninety degrees apart, and of such size that the split taken will be about 12.5 percent, have been arbitrarily chosen by the writers. A hollow 60° cone with an altitude of 3.5 inches and basal diameter of 4 inches was made. The cone was machined from stainless steel and polished to a mirror finish.

Figure 1 shows the equipment. An aluminum funnel, threaded (12 threads to the inch) into the horizontal holder, is used to direct the sample onto the apex of the cone. The funnel has a nominal half inch (0.508 inch) diameter hole at the bottom. The gap between the apex of the cone and the end of the funnel stem may be adjusted by turning the funnel. The post to the left of the funnel is marked in revolutions of the funnel (or the lead of the threads) and may be used as an indication of the gap opening. Each revolution of the funnel increases the gap by 0.041 inch or approximately one millimeter. A cup is used to catch the split and the major part of the sample which falls off the bottom edge of the cone is led away on a sloped curved surface which surrounds the sample cup.

Experience with a preliminary sheet metal model showed that material might clog in the funnel or that powders might not flow down the sides of the cone but slight tapping or vibrations alleviate these conditions. Accordingly, the entire splitter is mounted on a wooden box inside of which was attached an electric vibrator whose controls are mounted in front of the box. In operation, the unit is mounted on a level table.

The maximum capacity of the splitter depends on the retaining cup in which the sample is caught. The volume of the present cup is about 300 ml and the cup will hold 480 grams of 100 mesh quartz. As tests indicated that 13.5 percent of an assumed average sample would be captured, the amount of starting material could be 480/0.135 or 3.5 kilograms. This upper limit can be increased by increasing the volume of the retaining cup.

EXPERIMENTAL TESTS

The kind of test information desired needs consideration. The splitter, though designed specifically for crushed rocks passing 100 mesh, should be able to handle a variety of particle sizes in a minimum amount of time. Thus, there are three immediate questions to answer: (1) how much time is necessary for a split? (2) how reproducible is the splitting of an "average" rock sample passing 100 mesh? and (3) what is the effect of grain size on the amount of the split taken?

Estimates of possible effects due to the vibration would also be desirable. Inasmuch as the vibration is proportional to the current input to the vibrator, the control rheostat was calibrated by marking positions for different input currents. Positions corresponding to ammeter readings in 0.1 ampere from 0.6 to 1.0 ampere were used as one variable in the design.

Another factor that might affect the

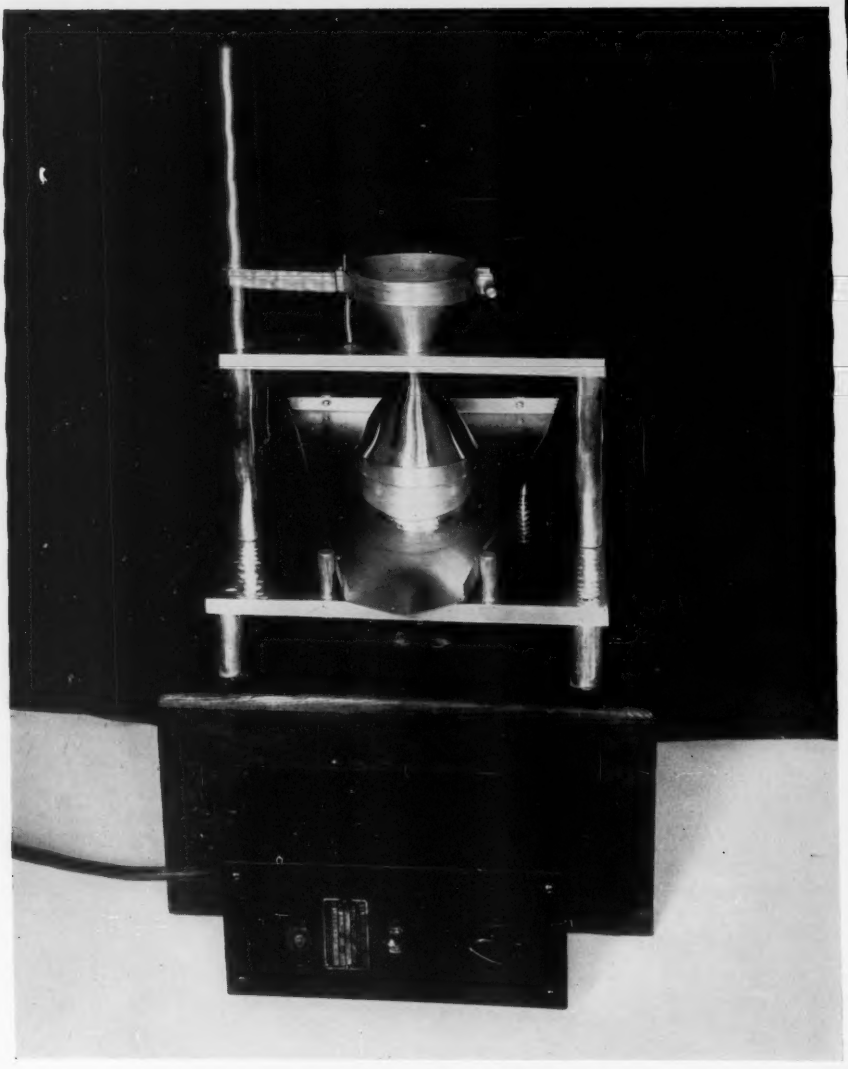

FIG. 1.—The slotted cone splitter.

amount of the split, and especially time required for operation, may be the rate of feed. This can be controlled by the gap between the cone and the tip of the funnel stem, and it can be measured in either millimeters or revolutions of the funnel as one revolution is equivalent to a one millimeter opening.

The amount of the sample taken by the splitter and the time required for the sample

to pass through can now be tested. For this test both the gap, measured in millimeters, and the vibration as measured by the current can be used as variables of classification. One hundred grams of an assumed "average" sample (quartz monzonite from Idaho) was used in the test so that the weights of the splits automatically represent percent as well as weight of the sample split. The approximate mineral composition of this sample is 35 percent of quartz, 30 percent each of orthoclase and plagioclase, 5 percent of mica (biotite and muscovite), and less than 1 percent of accessory minerals. The particle size analysis of this sample is shown in table 1.

Preliminary tests indicate that the time of passing through the splitter funnel and over the surface of the cone is largely a function of the rate of pouring the sample into the funnel. A Pyrex filter funnel was inverted in the sampler funnel to insure that the rate of pouring the sample would not be introduced as an uncontrolled variable. The filter funnel retained the sample in position, and the time required to pass through the system was expressed as the interval between removal of the filter funnel and passage of all the sample out of the splitter funnel. Preliminary tests also indicated that the vibration of the lowest current (0.6 ampere) was not sufficient to prevent the sample from caking and, hence, it did not flow through some of the gaps. Therefore, runs involving this current were omitted.

TABLE 1.—*Particle size analysis of a crushed quartz monzonite*

Mesh interval	Percent
− 60+ 80	1.6
− 80+100	4.5
−100+140	13.7
−140+170	33.6
−170+200	5.1
−200+230	7.9
−230	33.6

TABLE 2.—*Time (in minutes) required for splitting 100-gram sample of quartz monzonite*

Current (amp)	Gap (mm) 1	2	3	4	5
0.7	5.04	1.48	0.80	0.20	0.15
	4.43	1.64	0.82	0.25	0.16
0.8	4.01	1.53	0.45	0.16	0.17
	4.50	1.36	0.48	0.34	0.11
0.9	4.25	1.66	0.64	0.51	0.30
	3.28	1.42	0.99	0.51	0.22
1.0	3.19	1.48	0.70	0.40	0.27
	2.95	1.52	0.77	0.44	0.42

TABLE 3.—*Weight (or percent) of sample split*

Current (amp)	Gap (mm) 1	2	3	4	5
0.7	13.24	13.51	13.62	13.66	13.86
	14.31	12.91	13.22	12.83	13.47
0.8	13.52	14.41	13.37	13.08	12.31
	14.46	13.16	13.57	13.19	13.75
0.9	14.04	13.64	13.44	13.22	13.24
	13.77	13.92	13.45	13.21	13.23
1.0	13.65	13.85	13.36	12.95	13.69
	13.90	13.71	13.50	13.86	13.11

SPLITTING DATA AND CALCULATIONS

Observations were made in a previously determined random order. The time required for the sample to pour out of the splitter funnel was measured by an electric timer calibrated in hundredths of a minute and the weight of the split was obtained on a triple beam balance to the nearest 0.01 gram. The time required for splitting the 100-gram sample is shown in table 2, and the weight (or percent) of the sample split in table 3. The time required and the amount split for any determination occupy identical positions in tables 2 and 3, respectively. It is evident that the time required for splitting decreases as the gap increases and the time observations will not be discussed further.

PERCENT OF THE SPLIT

In actual operation the percent of the sample split is of main interest, but it would also be desirable to determine if there are significant effects when the observations are classified by either gap or current. Under the assumptions of normality and homogeneity of variance, calculations using the data of table 3 were then made as shown in Dixon and Massey (1951) and an analysis

TABLE 4.—*Analysis of variance*

Source of variation	SS*	DF*	MS*	F ratio
Current	0.0522	3	0.0174	
Gap	11.9492	4	.4873	
Interaction	1.0168	12	.0847	0.0847/0.2124<1 N.S.*
Subtotal	3.0182	19		
Within samples	4.2478	20	0.2124	
Total	7.2660	39		

* SS=sum of squares, DF=degrees of freedom, MS=mean square, N.S.=not significant.

TABLE 5.—*Analysis of variance*

Source of variation	SS	DF	MS	F ratio
Current	0.0522	3	0.0174	0.0174/0.1645<1 N.S.
Gap	11.9492	4	0.4873	0.4873/0.1645=2.96 S.*
Residual	5.2646	32	0.1645	
Total	7.2660	39		$F_{.95}(4, 32)=2.67$

* S.=significant.

of variance was obtained as shown in table 4.

The interaction was tested by the ratio, $F=0.0847/0.2124$, and as this ratio is less than the critical $F_{.95}(12,20)=2.28$, there is no evidence that the interaction is significant. The interaction and the within sums of squares and their degrees of freedom were pooled to obtain the new residual sum of squares shown in table 5. By forming F ratios with the mean squares, it is seen that the ratio of the current to residual is less than 1; and, hence, there is no statistical evidence that the current mean square is significantly larger than the residual. On the other hand, the mean square for the gap variable is significantly larger than the residual at the 5 percent significance level but but not at the 2.5 percent level. Hence the hypothesis that the gap opening has no effect on the percent of material sampled may be rejected at the 5 percent significance level, and it must be concluded that the opening has a significant effect.

As significant differences were obtained when the observations were classified by gap openings, the means for gaps 1 to 5 were calculated. These means together with their variances are shown in table 6. These data show in general that smaller openings give higher splitting yields. Although methods are available for comparing means in the analysis of variance (for example, Scheffe, 1953), a gap of three revolutions was chosen arbitrarily for future work because (1) the mean, 13.44, is very close to the grand mean of all the data, 13.50, and (2) the variance for this opening is a minimum for this set of data.

TABLE 6.—*Means and variances of splitting data classified by gap*

Gap (mm)	Mean	Variance
1	13.86	0.164
2	13.64	.214
3	13.44	.016
4	13.25	.120
5	13.33	.245

EFFECT OF PARTICLE SIZE

Another problem which frequently confronts those in geological work is the splitting of sized material. It is of interest therefore to determine whether the means of splits of different sized material are the same, or conversely, whether size affects the amount of split. In spite of the fact that vibration as qualitatively measured by current has no significant effect on the splitting of the previous sample, three currents were used to determine if current does affect the splitting of sized material.

The same design as in the previous sample was used, with five available sizes of magnetite as the sized samples, three currents as indications of vibration, and a constant opening of three revolutions (3 mm) as test conditions. The determinations were replicated 5 times with all runs made in random order. As before, the amount of sample split was 100 grams so that the weight split off automatically represented percent. The observations taken in the experiment (table 7) show the increase in the amount of sample split as the size of the grains decreases, but any effect of vibration is not apparent. To determine if there is an effect due to vibration, the two-way classification analysis of variance was made under the same assumptions as before and resulted in the analysis of variance shown in table 8.

As the interaction was not shown to be significant in the F test, the interaction and within sums of squares and their degrees of freedom were pooled to form the residual sum of squares shown in table 9.

As in the case of the crushed monzonite sample, vibration expressed as current shows nonsignificance by an F test, and as would be expected from an inspection of the data in table 7, the amount of sample split is dependent on the size of the material used.

ESTIMATING AVERAGE SPLITS

One of the problems in which the slotted cone splitter might be used is that of splitting large samples of sized materials for grain count analysis. In this type of splitting, it might be desirable to estimate beforehand the number of grains in the final split so that one would not be forced to count too many grains. Hence a calibration curve is necessary, or, assuming the function to be linear, a line of regression. Inasmuch as the factor of current has no significant effect on the amount split for the sized magnetite, all fifteen results in any size range were included in the calculations.

TABLE 7.—*Weight (or percent) of splits of sized magnetite*

Current (amp)	Mesh intervals				
	−20 +40	−40 +60	−60 +80	−100 +140	−170 +200
0.7	10.95	12.08	12.24	12.55	12.84
	11.23	12.47	12.38	12.63	13.15
	11.60	12.10	12.48	12.52	12.83
	11.38	12.08	12.35	12.56	12.86
	11.40	12.37	12.05	12.62	12.87
0.8	11.42	12.00	12.26	12.64	12.88
	11.14	12.05	12.41	12.45	12.78
	11.19	12.35	12.32	12.58	12.59
	11.41	12.16	12.05	12.58	12.80
	11.30	12.27	12.61	12.65	12.76
0.9	11.53	12.56	12.13	12.52	12.76
	11.48	12.08	12.33	12.53	12.80
	11.46	12.15	12.54	12.65	12.80
	11.53	12.21	12.37	12.82	12.71
	11.40	12.16	12.32	12.62	12.78

TABLE 8.—*Analysis of variance*

Source of variation	SS	DF	MS	F ratio
Current	0.0511	2	0.0255	
Size	18.5004	4	4.6251	
Interaction	.1495	8	.0187	0.0187/0.0209<1 N.S.
Subtotals	18.7010	14		
Within	1.2542	60	0.0209	
Total	19.9552	74		

TABLE 9.—*Analysis of variance*

Source of variation	SS	DF	MS	F ratio
Current	0.0511	2	0.0255	0.0255/0.0206=1.24 N.S.
Size	18.5004	4	4.6251	4.6251/0.0206=225 S.
Residual	1.4037	68	0.0206	
Total	19.9552	74		

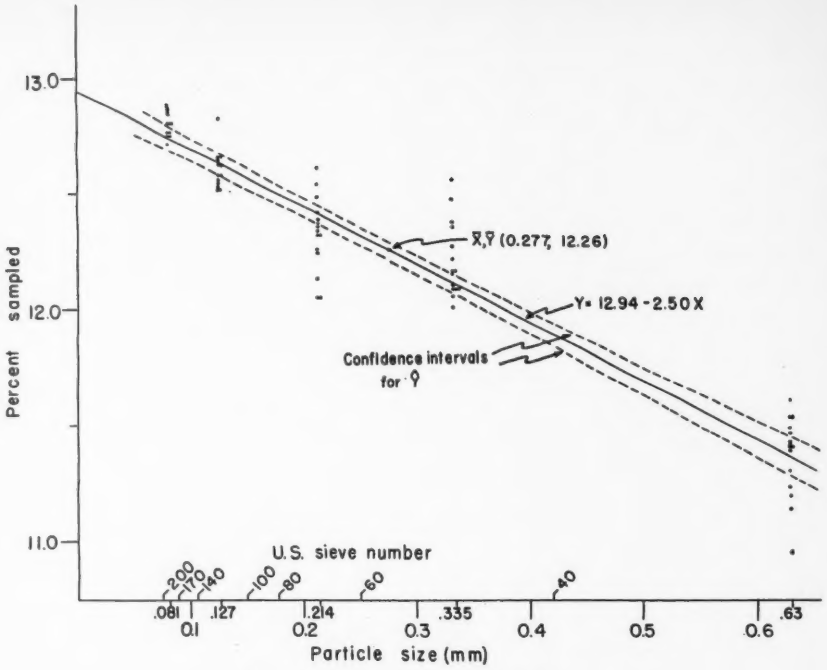

FIG. 2.—Percent sampled vs. particle size.

The scale for size ranges expressed as sieve numbers is nonlinear, so that the mean opening of the upper and lower sieves in each range was used as the mean diameter of the particles retained on any sieve. The percent sampled was then plotted against these mean diameters. These data are shown in figure 2.

It is assumed from the graph of the raw data that the relationship between the amount split and the mean sieve opening is linear and of the form $y=a+bx$, where y is the amount split, a is the intercept on the y axis, b is the slope of the line, and x is the mean diameter in mm. Least squares calculations yield the equation

$$y=12.94-2.50x \qquad (1)$$

as the line of regression of the amount split on the grain size.

A principal consideration in using a line of regression is the estimation of one variable, given the other variable, and the amount of confidence that can be placed in the estimation. Bennett and Franklin (1954, p. 228) have shown that confidence limits for the estimate, $\hat{y}$, of the average value of y, given any x_0, can be obtained by the formula:

$$(a+bx_0)-t_{n-2,\alpha}S_{y.x}\sqrt{\frac{1}{n}+\frac{(x_0-\bar{x})^2}{\Sigma(x-\bar{x})^2}}<\alpha+\beta x_0$$

$$<(a+bx_0)+t_{n-2,\alpha}S_{y.x}\sqrt{\frac{1}{n}+\frac{(x_0-\bar{x})^2}{\Sigma(x-\bar{x})^2}} \qquad (2)$$

where $\hat{y}$ has been replaced by its equivalent, $a+bx_0$.

If one substitutes a number of values of x_0 within the range of the x variable, one can obtain an upper and a lower limit at a significance level, α, for each estimate of a mean, $\hat{y}$, given any x_0. The 95 percent confidence intervals for several mean values are shown in table 10. By plotting these

limits and connecting each set of limits respectively with a smooth curve one can erect the 95 percent confidence limits for the averages around the line of regression as shown in figure 2.

With these confidence limits one may, within the range of the values in figure 2, estimate the average y value, $\hat{y}$, given any x value, x_0. In terms of the original variables, if one is given the size of the particles being split in the range of our variables, the average value of the amount sampled will occur between the upper and lower confidence limits 95 percent of the time.

The upper and lower confidence lines are for all practical purposes straight lines but do exhibit some curvature. In the more usual case where $S_{y \cdot x}$ the estimate of the standard deviation of the y observations and $t_{n-2,\alpha}$ are larger, the two confidence lines are hyperbolic in shape with vertices opposite each other at the point $(\bar{x},\bar{y})$. The distance between the two curves is a minimum at approximately the point $(\bar{x},\bar{y})$, indicating that the best estimates can be made in the vicinity of this point.

The average amount of the crushed quartz monzonite sampled was 13.50 percent whereas inspection of the graph (fig. 2) indicates that the greatest amount of sized material that can be sampled is 12.94 percent. When it is noted that about one third of the quartz monzonite passed a 230-mesh sieve, these two apparently contradictory results indicate the possibility of an interaction between the sizes of the crushed monzonite whereby the smaller sizes not only made their own specific contribution to the total amount split, but also interacted with the larger sized material so that the amount split was larger than that predicted by the line of regression. It would be expected, therefore, that if one sampled materials of different particle size compositions the average amounts sampled by the splitter would be different.

TABLE 10.—*Upper and lower limits for the average amount split, $\hat{y}$, given the grain size, x_0*

x_0	Upper limit	$\hat{y}$	Lower limit
0.65	11.41	11.32	11.23
.60	11.52	11.44	11.36
.50	11.75	11.69	11.63
.40	11.99	11.94	11.89
.35	12.10	12.06	12.02
.30	12.23	12.19	12.15
.25	12.36	12.32	12.28
.20	12.48	12.44	12.40
.15	12.60	12.56	12.52
.10	12.74	12.69	12.64
.075	12.80	12.75	12.70

ACKNOWLEDGEMENT

The work reported here is part of a program conducted by the U. S. Geological Survey on behalf of the Division of Raw Materials of the U. S. Atomic Energy Commission.

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JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 29, NO. 1, PP. 116–117
FIG. 1, MARCH, 1959

NOTE

AN INEXPENSIVE SAMPLE SPLITTER

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One of the problems facing those working in mineralogy and micropaleontology is the study of samples composed of large quantities of disaggregated microscopic mineral and test material. In order to limit laborious, time-consuming counting to a minimum it is necessary to isolate more easily handled fractions which will be statistically true representatives of the entire sample. The shortcomings of the quartering method, as a means of isolating these smaller units, are soon apparent to any investigator who has tried this procedure and, whenever available, most workers prefer the mechanical splitting devices for this purpose. However, frequently these are not available. The author recalls the frustrations experienced by students who because of either inaccessibility or lack of funds had to forego the use of a splitting device and use the quartering method in working with mineral or micro-fossil residues. Several years ago the laboratory of the Western Gulf Oil Company was unable to locate such a device and was therefore obliged to fabricate one using common materials found in any geologic laboratory. The constructed splitter has proven rugged enough to withstand constant use of over four years and was assembled at a negligible cost of time and material.

The construction of this miniature sample splitter is simple. An even number of standard petrographic slides is selected and each is cut along a diagonal. Scribing can be done with a common glass cutter, but a diamond-tipped pencil has proved to be more efficient. After cutting, the diagonal surface is ground smooth with fine-grit either on a lap or on a glass plate. These diagonally cut slides are placed so that the slopes of succeeding units will oppose (see sketch). Between diagonals regular, uncut slides are placed to act as slot separators. It is recommended that the exposed upper portions of the separators be beveled by grinding to prevent lodgment of grains while pouring.

Holes are drilled through the long dimension of each wood block and through each end of the two aluminum brackets and the device is ready for final assembly. The glass slides may or may not be cemented, for sufficient pressure to prevent loosening can be exerted by rotating the wood blocks at either side so the upper, inner edges press against the glass plates. Additional advantages of unglued plates are the facility of thorough cleaning and the replacement of damaged partitions.

TABLE 1.—*Specifications*

Overall dimensions: 60 mm long, 32 mm wide, 60 mm high.
17 petrographic slides: 46 mm long, 26 mm wide, 1.25 mm thick.
2 wood blocks: 26 mm long, 24 mm wide, 12 mm thick.
2 aluminum brackets: 50 mm long, 60 mm wide.
2 sets nuts, bolts, washers (bolts $1\frac{1}{2}$ inches long, $\frac{1}{8}$ inch diameter).

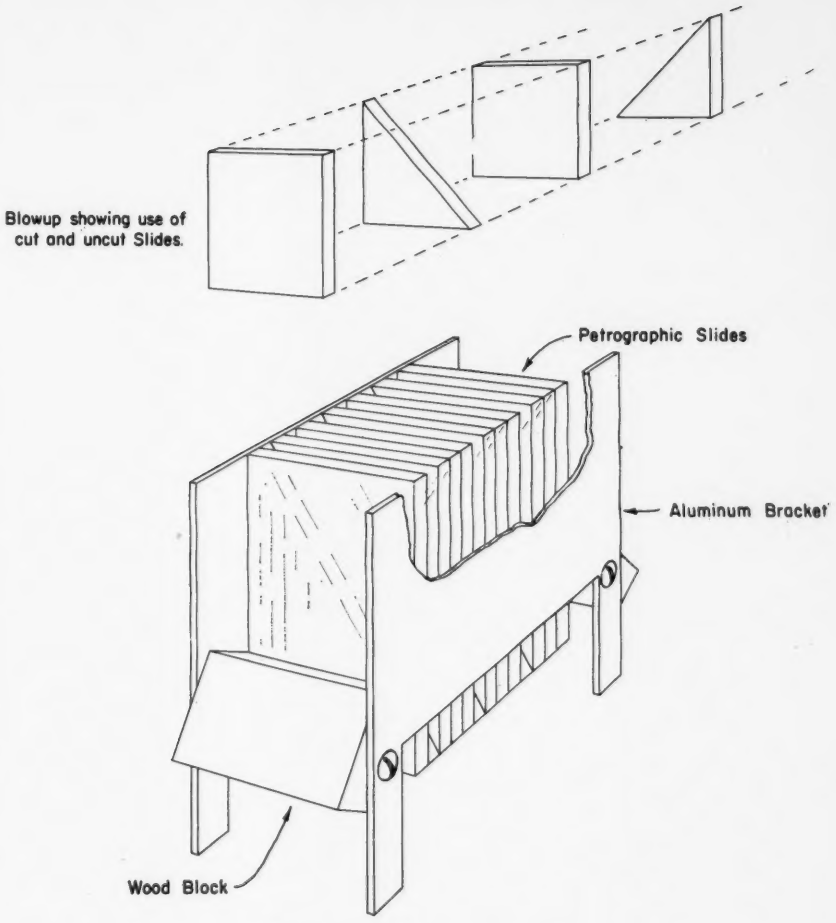

FIG. 1.—Assembly of sample splitter.

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DISCUSSION

IS PRIMARY DOLOMITE FAVORED BY HIGH OR LOW pH?[1]

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In one of his recent and, as usual, excellent articles, Dr. R. H. Dott, Jr. (1958, p. 8) states that, "Chilingar (1956) finds percentage of chert proportional to Ca/Mg ratio and precipitation of silica favored by decreasing pH (i.e. acidity). Primary dolomite is presumably favored by high pH and elevated temperature. If this be true, the very cherty nature of the Nevada Pennsylvanian limestones is consistent with their non-dolomitic character."

It is indeed gratifying to learn that my findings have been confirmed by Dr. Dott. These findings are also confirmed to some extent by experimental data. On varying the pH of sea water with Na_2CO_3, I found that the Ca/Mg ratio of direct precipitates decreases with higher pH (Chilingar, 1956, p. 32).

The statement of Dr. Dott that "Primary dolomite is presumably favored by high pH . . . , " however, is somewhat doubtful because it appears that the direct precipitation of dolomite out of sea water (primary dolomite) occurs at low pH (Bissell and Chilingar, 1958, p. 492).

Thus, a distinction should be made between the primary dolomites and the dolomitic limestones (namely, limestones high in Mg content). Possibly, this could explain why some primary dolomites contain abundant chert nodules and stringers. It is quite helpful to use the classification of limestones and dolomites on the basis of Ca/Mg ratio, with some divisions dictated by the ratios which are determined by the mode of formation (Chilingar, 1957, p. 187–189).

[1] Manuscript received January 22, 1959.

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JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 29, NO. 1, PP. 119–121
MARCH, 1959

REVIEWS

Geology of the Great Lakes, by Jack L. Hough, 1958. Pp. xviii + 313, 79 figs., 22 tables; 6×9 in., cloth. University of Illinois Press, Urbana, Ill. Price $8.50.

Part I, slightly less than half the book, depicts the modern lakes, geology of the basins, bottom topographies, level fluctuations, lake currents, temperature characteristics and overturns, chemistry of the waters, bedrock history, glacial history, and methods of dating.

Part II introduces history of the Great Lakes, recounts nature of evidence, beaches, deltas, etc., and some discussion of the postglacial uplift which has so altered the level of many features involved. Discussion of lake history ensues, for Erie and Huron, Lake Michigan, Superior, and Ontario basins, in separate chapters on each. For Algonquin and later lake stages, history of all basins involved are treated together, properly enough, in four chapters, on Lake Algonquin, post-Algonquin low stages, Nipissing Great Lakes, and transition from Nipissing to present. Though fewer stage names appear in these chapters, about as many pages are apportioned to them as to the total of all preceding stages in the several separate basins; and for good reason. This is the part of lakes history most radically revised since Leverett and Taylor's 1915 treatise; it was the time when nature was altering levels most radically, a time of low draining and of great uplift; also, it is this nearer approach to present, that most concerns archaeology and the distributions of lacustrine faunas.

The final chapter on radiocarbon chronology of Great Lakes History brings together available datings in the lake sequence, and is a fine reference for interested persons. It is to be hoped that this list will be much amplified during the coming decade, with more unequivocal material associated with the Algonquin beach, and for other stages.

Seventy-five good illustrations are included. A coordinated series of twenty-three maps depict the changing panorama of lake size, discharge, ice front, etc., and are probably the most extensive published set on the glacial Great Lakes. Even Monograph 53 omitted diagramming several of its stages.

A number of the innovations in Great Lakes history are the author's own. He has an observant eye for certain lacks (such as Nipissing beaches in southern Lake Huron), and relevant facts (Dreimanis' radiocarbon dates on site materials) and logic (no water, no downcutting) for dealing with them. And so the Nipissing and Algonquin levels are the same in southern Lake Huron. To the reviewer this looks well established.

Another novel interpretation is northward drainage from Erie to Huron basin during Grassmere, Lundy, and incipient Algonquin stages, and to an escape from Huron basin north, west, and southwest, via Mullet, Burt, Charlevoix and Torch Lakes, to Glacial Lake Chicago. A conclusion of Hough's to which this writer entirely subscribes is that the ice border during Algonquin beach stage was close to, and but little north of the Canadian Soo. However, his interpretation that the principal stage of Lake Algonquin was initiated by glacier advance closing the Kirkfield outlet, and was terminated by recession of ice reopening the same, after which the upper group beaches formed during down cutting of this outlet, militates against the following. The Algonquin beach has been traced into the Kirkfield-Fenelon Falls outlet at an elevation of 30–40 feet above outlet floor. This depth of water seems appropriate for a river that was as large as, or perhaps larger (as Taylor judged from examination of the outlet) than the present Port Huron drainway. But it seems nearly impossible that the Kirkfield outlet could accommodate the discharge of the same water body after it had dropped 40 feet in level below the Algonquin beach, as postulated by Hough's interpretation of the "upper group" beaches. Another item conflicting with this interpretation is Taylor's statement, subscribed to by R. E. Deane, that the "upper group of Algonquin beaches" converge down the tilt-line. This particular convergence is not well demonstrated, either by Taylor or by Deane, but without more factual evidence, the reviewer's impression (merely; but based on many field inspections) supports Taylor's.

The correlation of Niagara cutting of St. David's gorge with the Two Creeks low water stage, and the drift filling of this gorge during Valders readvance is interesting. Likewise is his inserting the Two Creeks low between earlier (upper) Lake Warren stages, and a later (lowest) Warren; he has the Wayne beach formed after "upper Warren," and after the Two Creeks low interval, but before the main "lowest Warren," which was a higher level than Lake Wayne. Leverett and Taylor simply had Lake Wayne preceding the higher Lake Warren. Later studies on river terraces by James W. Bay indicated a lake of Lake Wayne level, following Lake Warren. The reviewer suggests that contact with Bay's conclusions might modify, and even fortify, some of author Hough's ideas on this.

In line with his reinterpretation of Nipissing beach in southern Lake Huron, and for sake of down cutting by its southerly outflow being postponed until after Nipissing beach time, Hough seeks the point of Port Huron discharge attaining maximum only then and not before, *by denying any drainage from Lake Superior basin to the Port Huron outlet until Nipissing time.* Existing knowledge suggests otherwise. Leverett's (1929 a) strong beach (elevation 1000–1020 at south edge of Houghton Quadrangle) on Keweenaw Peninsula, 200 feet below, and parallel to, the Lake Duluth beach, even though it may not be the Algonquin, as Leverett thought, appears likely to associate with Lake Algonquin in time and in ultimate drainage course. Hough (p. 190) recognizes that this beach is too low to have connected with the St. Croix outlet (elevation 1017) which has been postglacially uplifted less than Houghton Quadrangle (by at least 100 feet). Also he has made a good case for the ice front in Lake Superior basin blocking the Au Train-Whitefish lowland during most of, possibly not all, Algonquin beach time. Until the Au Train-Whitefish passageway from Lake Superior to Lake Michigan basin might be shown *by former water levels* to postdate the Algonquin beach, and until the "upper group" (Algonquin) beaches are demonstrably dissociated from the Port Huron outlet, the evidence is against the author's italicized conclusion above. Although Lake Algonquin bordered the ice front near the present outlet of Lake Superior, and the Algonquin beach seems absent from the Superior basin immediately to the north, in Goulais Bay, nevertheless beaches of the upper group do occur at the latter place, at elevations up to 1060. Westward across the outlet moreover, at Dollar Settlement, on south shore of Whitefish Bay, there are beaches (elevation ca. 850–925), apparently of the upper group. It appears that Hough has over-exercised Leverett's evidence in quoting (p. 222) from him—"on the south shore of Whitefish Bay morainic contours extend down within 50 feet of the Lake Superior level, or 650 feet above the sea"—to support his own inference that wave action here reached no higher than this. Actually the strong beaches at Dollar Settlement, above mentioned, are only 1–2 miles southeast of such morainic contours (ca. 660–725), are 200 feet higher, and were formed by waves from the northwest quarter.

To rationalize the Algoma stage of the Great Lakes, Hough has ingeniously provided logical reasons for a temporary static level during the time of post-Nipissing uplift. There is good evidence on South Fox Island for believing the Algoma to have been a resurgent rather than a static water level, and it is alluded to briefly in R. T. Hatt's "Island Life in Lake Michigan." What caused the resurgence is enigmatic. It would require strong evidence to compel the repugnant conclusion of a temporary reversal in uplift for the northern lakes; and as Hough points out, this would not explain an Algoma beach in southern Lake Huron anyway. Perhaps it is worth examining whether, at this stage in post-Nipissing uplift, and because of uptilting, some large drainage became turned toward Lake Superior or northern Lake Huron, from a prior northward or northeastward course; and that this raised levels of the post-Nipissing lakes temporarily, and formed the Algoma beach before the rise was mitigated by outlet deepening and continued uplift.

Perhaps this is a good place for the reviewer to acknowledge debt to Mr. Hough for his bottom coring of Lake Michigan and bringing up so agreeable a substantiation of the "post-Algonquin low stages."

The reader should be aware that the book

brings up to date a complicated story, but that there is still much of Monograph 53 that is not "outdated" by it. It is best in presentation of revisions based on abundant new field data, but undoubtedly introduces some errors of its own in proposing revisions induced or deduced without enough new facts from the field. In illustrations it is far superior to the previous tome, and it seems to be more keenly analytical, but in very few spots does it convey an equivalent, ringing familiarity with things on the ground. The reader may enjoy as the reviewer did, the jolt of seeing Great Lakes precipitation tabulated in "cubic feet per second," with his own reminiscence of certain days when the Heavens burst. The reviewer, viewing reflection from his own conscience, admires this contribution which modernizes Great Lakes history, and highly values his copy of the book.

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A Treatise on Limnology, Vol. 1.—Geography, Physics, and Chemistry, by G. Evelyn Hutchinson, 1957. Pp. xiv+1015, 228 figs., 132 tables; $5\frac{3}{4}\times9$ in., cloth. John Wiley & Sons, Inc., New York, and Chapman & Hall, Ltd., London. Price $19.50.

It is unfortunate that the literal definition of "limnology" contains no clear implication of rivers or streams, for adherence to the literal meaning is apparently the reason that Hutchinson omits this recognized segment of his subject. Planned as a two-volume work, the completed project will deal exhaustively with lakes; the second volume is to cover limnobiology and the ecological, typological, and stratigraphic problems of lake development.

The book opens with chapters on the origin, morphology, and morphometry of lakes, then takes up the properties of water and the hydrological cycle and follows with hydromechanics and the optical and thermal properties of water. The last half of the volume is devoted to chemistry and covers inorganic ions, oxygen, carbon dioxide and pH, redox, nutrient cycles, minor elements, and organic matter. The book concludes with a long index of lakes and sixty-seven pages of bibliography containing about 1500 references heavily weighted toward the modern literature.

Hutchinson succumbs to the limnologists' old temptation to produce a classification of lakes. His classification into seventy-six types with several sub-types is at least geologically sound, as it is a classification of lake-forming processes. It will be of limited use to limnologists, for the quoting of a type-number can convey little of the characteristics of the aquatic environment.

The bipartite plan of the whole work is perhaps a little too cleverly pursued. The author cannot be criticized for obvious omissions from this volume because they may be planned for the next. For example, all discussion of sedimentation is omitted from the geological chapters of this volume. If sedimentation is included in the second volume, confusion will be unavoidable for it will then be in the biological section and in a certain sense out of context.

Hutchinson is admittedly a master of his craft and exhibits encyclopedic knowledge, but the inclusion of trivia such as the period of the seiche in a cup of coffee and the "path of gold" in Robert Browning's "Parting at Morning" lead this reviewer to question the judgment used.

The author has some writing habits which will eventually annoy the serious reader. He writes in a prose too frequently over-complex and over-elegant. He gives authoritative evaluations of the methods, data, or conclusions of others without indication that these are opinion. He consistently uses the word "empirical" for "observed," or some clear equivalent, and seems unaware of the automatic ambiguity with which the word's two definitions endow it. His policy of keeping his mathematical discussions at an esoteric level quite above that of the "non-mathematical novice" will not be appreciated by the novice who needs the mathematics.

Other reviewers have called this a great book. This reviewer will study it further before coming to a conclusion.

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JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 29, NO. 1, PP. 122–124
MARCH, 1959

ANNOUNCEMENTS

ANNUAL MEETING AND FIELD TRIP

TO BE HELD AT ALAMOGORDO, NEW MEXICO
APRIL 16, 17, & 18, 1959

The annual technical and business meeting of the Permian Basin Section, Society of Economic Paleontologists and Mineralogists will be held Thursday, April 16, 1959. Headquarters for the meetings and for the evening banquet will be the Desert Aire Motel, Alamogordo, New Mexico. General Chairman is M. Russell Stripp, Standard Oil Company of Texas, Box 746, Roswell, New Mexico. Chairman of the technical program is Richard F. Meyer, Humble Oil & Refining Company, Box 1287, Roswell, New Mexico.

A field trip on the west slope of the Sacramento Mountains, New Mexico, is planned for Friday and Saturday, April 17 and 18, 1959, under the leadership of Lloyd C. Pray, The Ohio Oil Company, Box 269, Littleton, Colorado. This trip will be sponsored jointly by the Permian Basin Section, Society of Economic Paleontologists and Mineralogists, and the Roswell Geological Society. Field Trip Chairmen are Carl Ulvog, Wilco Bldg., Midland, Texas, and Frank Packard, Box 1287, Roswell, New Mexico. Registration for the field trip will be on Thursday, April 16, 1959, at the Desert Aire Motel, Alamogordo, New Mexico.

SEDIMENTATION AND THE OIL INDUSTRY

A symposium, to be held in conjunction with the

FIFTH WORLD PETROLEUM CONGRESS

This Symposium is being scheduled for the morning and afternoon of June 1, 1959. It will be held in the Tudor Room of the Henry Hudson Hotel, 353 West 57th Street, just across the street from the New York Coliseum, from 9:15 A.M. to 12 noon and from 2:00 P.M. to 4:45 P.M.

Two general lectures will be given: the first on Recent sediments, in the morning and the second, on ancient sedimentation, in the afternoon. Each of them will be followed by a discussion period.

A series of general reports on various activities in the field of sedimentation, in various parts of the world, will be presented by the International Association of Sedimentology.

Further information may be obtained from Professor Francis P. Shepard, Scripps Institution of Oceanography, La Jolla, California, President of the Association, and also from Dr. André Vatan, Institut Français du Pétrole, Rueil (Seine & Oise), France, General Secretary of the Association.

A TABLE FOR THE CONVERSION OF MILLIMETERS TO PHI UNITS

JOHN C. GRIFFITHS AND DONALD D. MCINTYRE

Published by the Mineral Industries Experiment Station, College of Mineral Industries, The Pennsylvania State University (May, 1958), 56 pp., $8\frac{1}{2} \times 11$ in., $1.60 including postage. Sold by the Department of Mineralogy, The Pennsylvania State University, University Park, Pennsylvania. The table was prepared by use of the Pennstac computer. It extends from minus 14 to plus 14 phi units.

PALEONTOLOGICAL AND MINERALOGICAL ASPECTS OF POLAR WANDERING AND CONTINENTAL DRIFT—A SYMPOSIUM
ATLANTIC CITY, APRIL, 1960

As announced in the last issue of the *Journal of Sedimentary Petrology*, the Society of Economic Paleontologists and Mineralogists is sponsoring a symposium on the paleontological and mineralogical aspects of polar wandering and continental drift at their annual meeting in Atlantic City in April, 1960. Already the following outstanding speakers have accepted invitations to present papers at the symposium:

Geo. W. Bain, Department of Geology, Amherst College, Amherst, Mass.

S. Warren Carey, Department of Geology, University of Tasmania, Hobart, Tasmania.

Kenneth E. Caster, Department of Geology, University of Cincinnati, Cincinnati, Ohio.

Ernst R. Deutsch, Imperial Oil Limited, Calgary, Alberta, Canada.

Maurice Ewing, Lamont Geological Observatory, Palisades, N. Y.

Horacio J. Harrington, Tennessee Gas Transmission Co., Houston, Tex.

S. Keith Runcorn, Physics Department, King's College, Newcastle upon Tyne, England.

There will be ample opportunity for full discussion of this problem by anyone interested, as an entire day—morning, afternoon, and evening—will be devoted to the subject. Scientific thought is still poles apart, even within the geological profession. It is suggested that we pool our knowedge, listen to what others have to say, and see if we cannot resolve the differences. Those interested in contributing to the program or discussions are urged to contact the chairman of the symposium, Dr. A. C. Munyan, Vice Chairman, Research Committee,, S.E.P.M., Sohio Petroleum Company, Box 2558, Billings, Montana.

In the face of new evidence, the geological profession is urged to reconsider the concept of crustal shifts. There is still no better method than Chamberlin's multiple working hypothesis, and there is absolutely no valid evidence for ruling out crustal shifts or relative movements of continents. In this respect, the following statements offer a challenge:

"There is evidence for a relative movement of 1000 miles between America and Europe since Triassic times." (Creer, *et al.*, 1957)

"The magnetizations of over 400 basalt specimens from the Deccan Traps (of India) have been found to be aligned in general along a NNW to NW axis dipping towards the south. The results are consistent with the supposition that India had drifted northwards through over 50° of latitude and rotated 25° anticlockwise within the last 70 million years.

"Ancient pole positions corresponding to these results differ considerably from those obtained by other workers for contemporaneous European and North American rocks, and this suggests that part of the postulated movement of India has been in relation to other continents." (Deutsch, *et al.*, 1958)

"The face of the globe shows many areas where orogenic belts wheel in trend through large angles, sometimes as much as 180°. Such a form could have one of two origins. Either the orogenic zone had that shape from the beginning, or the bend represents an impressed strain (. . . defined as an *orocline*). Classical geology has always made the first assumption, explicitly or implicitly, using the concept of cratons around which the oroclines were moulded. However, logical scientific analysis demands that we should also examine the consequences of making the second assumption. . . . When all such oroclines, together with other identifiable strains, are reversed, there appears a Laurasia substantially identical with that deduced by Du Toit on wholly different grounds." (Carey, 1955)

In a recent paper (Gussow, 1958), the term metastasy (mĕ.tăs'tà.sĭ), to shift from place to place, shift of the crust [from Greek *metastasis*, to place in another way; fr. Gr. *meta* change, transposition, transfer, shift+*histanai* to place], was introduced for horizontal adjustments of the crust, in contrast to isostasy for vertical adjustments. Gussow is convinced that not only has the crust shifted repeatedly with respect to the interior, but relative displacements of segments of the crust have also occurred.

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ERRATA

NOTES ON DIAGENETIC DOLOMITIZATION

HAROLD J. BISSELL AND GEORGE V. CHILINGAR, 1958, *Jour. Sedimentary Petrology*, v. 28, p. 490–497

In figure 1, p. 491, blocks 8 and 15 should be solid black, whereas block 14 should be crosshatched as in block 17. Strakhov made this mistake on his original graph, but he corrected it in a later article. The present writers' draftsman mistakenly used the original graph as a basis for figure 1.

On page 495, right-hand column, the sentence beginning below the formula should read, "The reaction goes to the right in the initial stages . . . " (instead of "left").

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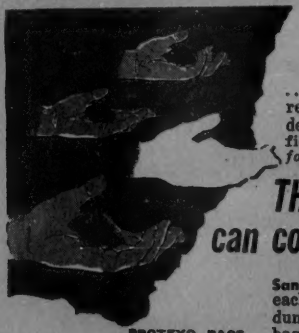

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